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VOL IV OF V

ASSESSMENT OF  
ENVIRONMENTAL CONTAMINATION  
EXPLORATORY STAGE  
TOOELE ARMY DEPOT  
TOOELE, UTAH

VOLUME IV APPENDICES F THROUGH I

IRP 81-04



*The Earth Technology Corporation*

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ENVIRONMENTAL CONTAMINATION  
EXPLORATORY STAGE  
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**VOLUME IV APPENDICES F THROUGH I**

**APPENDIX F: SEMI-QUANTITATIVE ANALYTICAL METHODS  
APPENDIX G: QUALITY CONTROL REPORTS  
APPENDIX H: QUALITY CONTROL RESULTS  
APPENDIX I: SUMMARY OF QUALITY CONTROL DATA AND  
FIELD SAMPLE DATA**

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**TOOELE ARMY DEPOT  
PROCUREMENT DIVISION  
P.O. BOX D  
TOOELE, UTAH 84074  
AND  
U.S. ARMY TOXIC AND HAZARDOUS  
MATERIALS AGENCY  
ABERDEEN PROVING GROUND, MARYLAND 21010**

**Prepared by:**

**KEVIN T. BLOSE  
ERIC G. LAPPALA  
ROBERT L. STOLLAR  
KAREN F. KNIRSCH**

**ERTEC WESTERN, INC.  
3777 LONG BEACH BOULEVARD  
LONG BEACH, CALIFORNIA 90807**



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## F-1.0 ANALYSIS OF ANIONS IN WATER BY ION CHROMATOGRAPHY

- I. Application: This method is applicable to the qualitative and semi-quantitative analysis of the following ions:

Chloride ( $\text{Cl}^-$ )  
 Fluoride ( $\text{F}^-$ )  
 Nitrate ( $\text{NO}_3^-$ )  
 Nitrite ( $\text{NO}_2^-$ )  
 Phosphate ( $\text{PO}_4^{-3}$ )  
 Sulfate ( $\text{SO}_4^{-2}$ )

- A. Tested Concentration Range: 1 mg/liter to 5 mg/liter for each analyte.

- B. Sensitivity: Peak height in mm at detection limit.

$\text{Cl}^-$	-	62 mm	$\text{NO}_2^-$	-	25 mm
$\text{F}^-$	-	159 mm	$\text{PO}_4^{-3}$	-	7 mm
$\text{NO}_3^-$	-	8 mm	$\text{SO}_4^{-2}$	-	8 mm

- C. Detection Limit (mg/l):

$\text{Cl}^-$	-	1	$\text{NO}_2^-$	-	0.9
$\text{F}^-$	-	1	$\text{PO}_4^{-3}$	-	0.8
$\text{NO}_3^-$	-	1	$\text{SO}_4^{-2}$	-	1

- D. Interferences: Sulfite, if present, may co-elute with nitrate. In natural water samples it is anticipated that sulfite will have oxidized to sulfate.

- E. Analysis Rate: An analyst can analyze 15 samples per day after instrument calibration.

- II. Chemistry: Analytes are ionic species in a water solution, standards for the analysis are prepared from the sodium and potassium salts as indicated below.

III. Apparatus:

- A. Instrumentation: Dionex Model 10 Ion Chromatograph equipped with the following columns:

precolumn - 4 x 50 mm anion precolumn  
 separator - 4 x 250 mm anion separator  
 suppressor - 9 x 100 mm anion suppressor

**B. Parameters:**

1. Eluent - 0.003 M  $\text{NaHCO}_3$ /0.0024 M  $\text{Na}_2\text{CO}_3$  @ a flow rate of 138 ml/hr (30% pump capacity)
2. Columns - noted above
3. Injection volume - 100  $\mu\text{L}$
4. Recorder Speed 30 cm/hr using a 1 volt input strip chart recorder
5. Conductivity meter setting 10  $\mu\text{mHO/cm}$  full scale

**C. Hardware/Glassware:**

1. Glass and polypropylene volumetric flasks for the preparation of stock and standard solutions (1000 mL and 100 mL sizes).
2. Class A transfer pipettes for preparing standards (10, 20, 30, 50, and 100 mL sizes).
3. Syringes, 10 mL, polyethylene with luer tip.
4. In-line filter holders (Swinnex-type, 25 mm)
5. 25-mm membrane filters, 0.45  $\mu\text{m}$  pore size.

**D. Chemicals:**

1. Sodium Carbonate
2. Sodium Hydrogen Carbonate
3. 1 N Sulfuric Acid for column regeneration
4. Sodium Fluoride
5. Sodium Chloride
6. Sodium Nitrate
7. Sodium Nitrite
8. Dibasic Sodium Hydrogen Phosphate dodecahydrate ( $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ )
9. Potassium Sulfate

**IV. Standards:**

- A. Calibration Standards: (Standards will be prepared from or checked against SARMS.)

To obtain 1000  $\mu\text{g/mL}$  stock solutions, dissolve the following amounts of reagent grade chemicals in deionized water:



1.  $F^-$  - 3.0579 g KF/liter
2.  $Cl^-$  - 2.1032 g KCl/liter
3.  $NO_3^-$  - 1.6308 g  $KNO_3$ /liter
4.  $NO_2^-$  - 1.4998 g  $NaNO_2$ /liter
5.  $PO_4^{3-}$  - 1.2112 g  $NH_4H_2PO_4$ /liter
6.  $SO_4^{2-}$  - 1.4792 g  $Na_2SO_4$ /liter

With the exception of the phosphate salt, the other five chemicals will be oven-dried @ 105°C for one half hour and cooled to room temperature in a desiccator prior to weighing. The  $NH_4H_2PO_4$  should be dried for two hours at 110°C.

From the above stock solutions, mixed working standards in three concentration ranges will be prepared in volumetric flasks:

Table 1. Preparation of Standard Solutions for Instrument Calibration

Anion	High Range Standard			
	mL of each Stk. Sol. (1.00 mL=1.00 mg) Diluted to one liter	High Range Std. µg/mL	Intermediate Range Std. µg/mL	Low Range Std. µg/mL
Fluoride ( $F^-$ )	20	20	2.0	0.4
Chloride ( $Cl^-$ )	20	20	2.0	0.4
Nitrite ( $NO_2^-$ )	20	20	2.0	0.4
Phosphate ( $PO_4^{3-}$ )	20	20	2.0	0.4
Nitrate ( $NO_3^-$ )	20	20	2.0	0.4
Sulfate ( $SO_4^{2-}$ )	20	20	2.0	0.4

1. Prepare a high range standard solution by diluting the volumes of each anion specified in Table 1 together to 1 liter with deionized water.
2. Prepare the Intermediate Range Standard Solution by diluting 10.0 mL of the High Range Standard Solution (see Table 1) to 100 mL with deionized water.
3. Prepare the Low Range Standard Solution by diluting 20.0 mL of the Intermediate Range Standard Solution (see Table 1) to 100 mL with deionized water.

B. Control Spikes:

1. Prepare a spiking solution by adding 1 mL of each calibration stock (1000 ppm) to a 100 mL volumetric flask, and dilute to volume. Concentration of spiking solution is 10  $\mu\text{g/mL}$ .
2. Spike a water matrix with the spiking solution as indicated below.

<u>mL water</u>	<u>mL spike</u>	<u>conc. of spike (for all analyte)</u>
10	0	0
9.50	0.50	0.5 $\mu\text{g/mL}$
9.00	1.00	1 $\mu\text{g/mL}$
8.00	2.00	2.0 $\mu\text{g/mL}$
5.00	5.00	5 $\mu\text{g/mL}$
0	10.00	10 $\mu\text{g/mL}$

V. Procedure;

- A. Prepare ion chromatograph eluent by dissolving 5.0400 grams  $\text{NaHCO}_3$  and 5.0880 grams  $\text{Na}_2\text{CO}_3$  in 20 liters of deionized water. This solution is stored in a 5 gallon carboy and 4 liter aliquots are taken in collapsible bottles and placed in the Dionex for use.
- B. The ion chromatograph detector is checked for zero and full-scale deflections with deionized water flowing through the system. After obtaining a stable water baseline as monitored by the stripchart recorder, eluent is allowed to flow through the columns until equilibrium is established (again indicated by the establishment of a stable baseline). Normally, one-half to one hour is adequate to set up equilibrium.
- C. The volumes of the aqueous samples will be measured to 0.1 ml by using graduated cylinders.
- D. All samples and natural water blanks are filtered through 0.45  $\mu\text{m}$  pore size membrane filters located in the Swinnex filter holder, prior to injection into the ion chromatograph.
- E. The analysis scheme consists of running the prepared standards first followed by 5 samples and the blank and repeating this order until all samples have been analyzed. The suppressor column requires regeneration every 12-14 hours with the dilute sulfuric acid. Using fast-run columns, a sample can be injected every 10 minutes.

## VI. Calculation:

- A. Standard Curve: Peak heights for each standard are measured on the chromatogram, and a plot of peak height versus concentration generated for each analyte.
- B. Concentrations observed for each sample are obtained by comparing measured peak heights from the chromatogram with the standard curve for each analyte.

## VII. References:

1. Dionex Model 10 operating manual, Marvin J. Fisherman, Grace Pyen.
2. "Determination of Selected Anions in Water by Ion Chromatography," U.S. Geological Survey, September 1979.
3. USATHAMA Method 2P.

## F-2.0 ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN WATER BY GC-MS

- I. Application: This method is used for the semiquantitative determination of volatile organic compounds in water and is based on EPA Method 624 (see reference section). Compounds used to validate the performance of this method are listed below. The user should consult EPA Method 624 for information concerning the applicability of this method for other compounds.

A. Tested Concentration Range - ( $\mu\text{g/L}$ )

Benzene	0.58 to 11.6
Bromomethane	0.52 to 10.4
Chlorobenzene	0.47 to 9.5
1,2-Dichloroethane	0.54 to 10.8
Trans-1,2-Dichloroethene	0.54 to 10.8
1,1,2,2-tetrachloroethane	0.56 to 11.2
1,1,1-trichloroethane	0.53 to 10.6
Trichloroethene	0.60 to 11.9

- B. Sensitivity - The integrated area response for the most intense fragment ion of each compound at 1  $\mu\text{g/L}$  a 5.0 mL water sample is presented below;

Most Intense Mass Spectral Compound	Fragment Ion	Ion Count
Benzene	78	1470
Bromomethane	94	240
Chlorobenzene	112	460
1,2-Dichloroethane	62	240
Trans-1,2-Dichloroethene	96	320
1,1,2,2-tetrachloroethane	83	370
1,1,1-trichloroethane	97	260
Trichloroethene	130	320

C. Detection Limit:  $\mu\text{g/L}$ 

Benzene	2
Bromomethane	4
Chlorobenzene	2
1,2-Dichloroethane	2
Trans-1,2-Dichloroethene	2
1,1,2,2-tetrachloroethane	2
1,1,1-trichloroethane	2
Trichloroethene	3

- D. Interferences - Compounds which coelute and have similar mass spectra to the compounds of interest may interfere.
- E. Analysis Rate - Approximately 1.1 hours are required to spike, purge, desorb, and analyze a sample for GC-MS analysis. One analyst can analyze 7 samples in an 8-hour day.

## II. Chemistry

### A. Alternate Nomenclature and Chemical Abstracts Registry Number

Benzene:	$C_6H_6$ Benzol; Benzole; Phene CAS RN 71-43-2
Bromomethane:	$CH_3$ Br Methyl Bromide; Monobromomethane CAS RN 74-83-9
Chlorobenzene:	$C_6H_5Cl$ Benzene Chloride; Monochlorobenzene CAS RN 108-90-7
1,2-Dichloroethane:	$C_2H_4Cl_2$ Ethylene Chloride; Ethylenedichloride; Dutch Liquid CAS RN 107-06-2
Trans-1,2-Dichloroethene:	$C_2H_2Cl_2$ Dichloroethylene; Acetylene Dichloride CAS RN 156-59-2
1,1,2,2-tetrachloroethane:	$C_2H_2Cl_4$ Acetylene Tetrachloride CAS RN 79-34-5
1,1,1-trichloroethane:	$C_2H_3Cl_3$ Methylchloroform; Chloroethene CAS RN 71-55-6
Trichloroethene:	$C_2HCl_3$ Trichloroethylene; Ethinyl Chloride CAS RN 79-01-6

### B. Physical and Chemical Properties

	Melting Point (°C)	Boiling Point (°C)	Density	Volume/ Weight
Benzene	5.5	80.1	$d_4^{15}$ 0.8787	22.8 $\mu$ L/20 mg
Bromomethane	-93.66	3.56	$d_{gas}^{20}$ 3.974 g/L $d_4^0$ 1.730	11.6 $\mu$ L/20 mg
Chlorobenzene	-45	132	$d_4^{20}$ 1.1064	18.1 $\mu$ L/20 mg
1,2-Dichloroethane	-35	84	$d_4^{20}$ 1.256	15.9 $\mu$ L/20 mg
Trans-1,2-Dichloroethene	-50	47.5	$d_4^{20}$ 1.2565	15.9 $\mu$ L/20 mg

1,1,2,2-tetrachloroethane	-43.8	146	$d_{4}^{20}$ 1.5984	12.5 $\mu$ L/20 mg
1,1,1-trichloroethane	-33	74	$d_{4}^{20}$ 1.3492	14.8 $\mu$ L/20 mg
Trichloroethene	-88	87	$d_{4}^{20}$ 1.462	13.7 $\mu$ L/20 mg
Bromochloromethane	-86.5	69	1.991 <sup>19</sup>	100.5 $\mu$ L/200 mg
2-Bromo-1-Chloropropane		117.0 <sup>756</sup>	$d_{4}^{20}$ 1.537	130.1 $\mu$ L/200 mg
1,4-Dichlorobutane	-38.7	161-163	$d_{4}^{20}$ 1.1598	172.4 $\mu$ L/200 mg

Data on Bromomethane from Merck Index, 9th Ed. All other data from the Handbook of Physics and Chemistry, 48th Ed.

Use caution in handling the compounds listed above. Potential toxic inhalation and skin absorption hazard exist.

C. Chemical Reactions - N/A

III. Apparatus:

A. Instrumentation - Hewlett Packard 5985 gas chromatograph and mass spectrometer, or equivalent, equipped with an all glass jet separator and interfaced with a gas chromatograph with an all-glass, on-column injector system designed for packed column analyses. The system must be equipped with an appropriate data system to allow acquisition of full mass range scans for the duration of the chromatographic run. The computer should be equipped with mass storage devices for saving the data from the GC-MS. There should be computer software available to allow searching of any GC-MS run for specific ions and plotting the ions with respect to time or scan number. The ability to integrate the area under any specific ion plot is essential for quantification. A Valco purge and trap device matched with a Tekmar autosampler or equivalent equipped with 5.0-mL fritted purging devices, a sorbent trap consisting of a 12 inch x 1/8 inch ID steel trap packed with 2/3 Tenax GC (60/80 mesh) and 1/3 Davison Type-15 silica gel (35/60 mesh).

B. Parameters

1. Column - 1% SP-1000 on 60/80 mesh Carbopak B; 1/4 inch x 2 mm ID x 6 foot glass column.
2. Gas flow - 35 mL/min Helium
3. Temperatures - Injector - 240°C  
 Source - 200°C  
 Oven - 35°C hold 2 minute;  
       - 8°C per min  
       - 220°C hold 26 minute

4. Sample volume - 5 mL
5. Detector - mass spectrometer scanned from 42-342 amu every second for the first 24 minutes of the run, and 42-542 amu every second for the last 26 minutes for the run.

6. Retention times -

Benzene	20.0 min
Bromomethane	5.2 min
Chlorobenzene	29.0 min
1,2-Dichloroethane	15.1 min
Trans-1,2-Dichloroethene	14.0 min
1,1,2,2-tetrachloroethane	25.4 min
1,1,1-trichloroethane	16.5 min
Trichloroethene	19.6 min
2-Bromo-1-Chloropropane	21.7 min
Bromochloromethane	12.6 min
1,4-Dichlorobutane	25.8 min

C. Hardware/Glassware

1. Syringe - 5.0-mL calibrated glass with Teflon removable plunger and shut-off valve (e.g., Glenco 19925 Series Gas/Liquid Syringe with No. 925-MV Valve)
2. Microsyringes - 10- $\mu$ L, 20- $\mu$ L, 50  $\mu$ L, 100- $\mu$ L, 250- $\mu$ L
3. Alarm clock
4. 10 mL ground glass stopped volumetric flask

D. Chemicals

1. Methanol, distilled in glass
2. Water, laboratory-grade, organic free
3. SARMS or INTERIM SARMS
4. p-Bromofluorobenzene - reagent grade
5. Internal Standards - 2-bromo-1-chloropropane, bromochloromethane, and 1,4-dichlorobutane

E. Reagents - N/A

IV. Standards

A. Calibration Standard

1. Individual Stocks (2 mg/mL) - Prepare individual stock solutions in methanol according to EPA Method 624 by placing 9.8 mL of methanol into a 10 mL ground glass stoppered volumetric flask, weighing, adding 20 mg (see Section IIB for volumes) of assayed reference liquid from a 50  $\mu$ L syringe, and reweighing. (See Physical and Chemical Properties for volume to weight relationships.)

2. Stock Calibration Mixture (5 ng/ $\mu$ L) - Using a 50  $\mu$ L syringe place 25  $\mu$ L of each Individual Stock in a 10 mL volumetric flask containing 5 mL of methanol and dilute to mark with methanol. Mix thoroughly, transfer to screw capped tube (Teflon liner), and store refrigerated at -20°C.
3. Stock Calibration Mixture (1 ng/ $\mu$ L) - Using a 10  $\mu$ L syringe place 5  $\mu$ L of each Individual Stock (2 mg/mL) in a 10 mL volumetric containing 5 mL of methanol and dilute to the mark with methanol. Mix thoroughly, transfer to (Teflon-lined) crew-capped tube, and store at -20°C.
4. Internal Standard Stock (20 mg/mL) - Using a 250  $\mu$ L syringe place as described above in A.1. 200 mg each of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane in a 10 mL volumetric flask containing 5 mL of methanol and dilute to 10 mL with methanol. (See Section IIB for volumes.) Mix thoroughly and store at -20°C.
5. Diluted Internal Standard (4 ng/ $\mu$ L) - Using a 20  $\mu$ L syringe place 20  $\mu$ L of Internal Standard Stock into 100 mL of organic free water and mix thoroughly. Store in a screw capped bottle and prepare fresh each day. Before each sample is analyzed 25  $\mu$ L of this Diluted Internal Standard is added to the 5 mL syringe.
6. Working Calibration Standards - Standards are prepared according to the scheme shown below.

<u>Concentration of Analytes in Aqueous Sample</u>	<u>Volume (<math>\mu</math>L) of Calibration Standard to Add to 50 mL Water Sample</u>
0	0
0.5 $\mu$ g/l	25 of 1.0 ng/ $\mu$ L stock soln.
1.0 $\mu$ g/l	50 of 1.0 ng/ $\mu$ L stock soln.
2.0 $\mu$ g/l	20 of 5.0 ng/ $\mu$ L stock soln.
5.0 $\mu$ g/l	50 of 5.0 ng/ $\mu$ L stock soln.
10.0 $\mu$ g/l	100 of 5.0 ng/ $\mu$ L stock soln.
20.0 $\mu$ g/l	200 of 5.0 ng/ $\mu$ L stock soln.

Methanolic calibration standards should be prepared weekly from stock solutions and stored at -20°C. Aqueous standards should be prepared daily.

7. Stock Instrument Tuning Standard (2 mg/mL) - Prepare solution (2 mg/mL) of p-bromofluorobenzene (13.3  $\mu$ L/20mg) as described in Part IV.A.1.



8. Diluted Instrument Tuning Standard (10  $\mu\text{g/mL}$ ) - Using a 100  $\mu\text{L}$  syringe place 50  $\mu\text{L}$  of Stock Instrument tuning standard in 10 mL volumetric flask and dilute to 10 mL with methanol.
- B. Control Spikes - The control spike experiments will be performed using the same solutions used for working calibration standards and at the same levels.
- V. Procedure: The sample for volatile organic compound analysis should be collected in a 40-mL vial which is filled to overflowing, sealed with a Teflon-lined cap, and stored at 4° C in the inverted position. The samples should be analyzed within 7 days of collection.

Prior to injecting any calibration standards onto the GC-MS, insure correct operation of the GC-MS system by injecting 5.0  $\mu\text{L}$  of the solution containing 10  $\mu\text{g/mL}$  of p-bromofluorobenzene (BFB). The correct ion balances are shown in Table 2.

TABLE 2. CORRECT ION ABUNDANCES OF BFB TO ENSURE PROPER TUNING AND SENSITIVITY OF THE MASS SPECTROMETER

<u>Mass</u>	<u>Abundance</u>
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 to 9% of mass 174
176	> 95% but < 101% of mass 174
177	5 to 9% of mass 176

The relative sensitivity of the instrument is determined by measuring the area of m/e 95 daily and recording the area counts on a control chart.

A hard copy of the BFB mass spectrum should be included with the mass spectra of reference and sample mass spectra acquired during the day's analyses. Inject the BFB standard at 220°C and run isothermally. The retention time of BFB under these conditions is about 18 minutes. Analyze a method blank consisting of organic free water spiked with the 3-component internal standard solution. Pour the water into the syringe with the valve closed and the plunger removed. Replace the plunger, open the valve and expel the water sample with the syringe valve facing upward until exactly 5.0 mL remain in the syringe. Open the valve and spike the water sample with internal standards by inserting the spiking syringe needle directly through the open sample syringe valve and injecting 25 µL of the 4 ng/µL Dilute Internal Standard Solution. Transfer the sample to the purging device. Purge the sample with purified helium or nitrogen for 10 minutes at 44 mL/min. The adsorption trap is automatically switched into the GC carrier flow and heated to 180°C for 5 minutes with the GC cryogenic cooling on and the column at about 35°C. Start GC-MS data acquisition at the beginning of the heat desorption period. After 2 min. start the GC temperature program at 8°C/min to 220°C. After 5 min. the adsorption trap is automatically switched back into the purging gas flow and baked out by heating to 180°C for 15 minutes, then cooled for 10 minutes.

Be sure that none of the compounds of interest are present in the method blank sample at levels greater than the lower limit of detection prior to analysis of the calibration standards. Analyze 3 calibration standards bracketing the range of interest (usually 1, 2, and 5 µg/L). Obtain a clean method blank at the beginning of each day prior to analysis of any calibration standard or samples. For each run determine the area under the peak of the characteristic fragment ion at the correct retention time for each compound of interest.

VI. Calculations: For each of the calibration standards determine the mass spectrometric response factor according to the following equation:

$$\text{Response Factor } RF = \frac{(A_s) (C_{is})}{(A_{is}) (C_s)}$$

Where

$A_s$  is the integrated area of the characteristic ion for the pollutant standard.

$A_{is}$  is the integrated area of the characteristic ion of the internal standard.

$C_{is}$  is the amount ( $\mu\text{g}$ ) of the internal standard injected.

$C_s$  is the amount ( $\mu\text{g}$ ) of the analyte standard injected.

Use the internal standard eluting nearest the compound of interest as the reference internal standard for determining response factors. The response factors should be relatively constant over the entire concentration range tested. Determine the mean, standard deviation, and percent relative standard deviation of the response factors. If the percent relative standard deviation of the response factors is no more than 20% use the mean response factor for each compound. If the response factors vary significantly with concentration, prepare a calibration curve where the RF is plotted against the standard concentration. Once this calibration curve has been determined it should be verified daily by injecting at least one standard solution. Calculate the concentration according to the following equation using the appropriate response factor from the calibration curve.

$$C = \frac{(A_s) (C_{is})}{(A_{is}) (RF) (0.005 \text{ L})}$$

where C is the sample concentration in  $\mu\text{g}/\text{l}$ .

Use the same equation to quantify each compound of interest in the samples using the mean response factor or appropriate response factor from the calibration curve estimated from the area counts of the compound of interest relative to the appropriate internal standard. The correct internal standard to be used for quantification of each compound is given in Table 3. Additional criteria for qualitative and quantitative determination of organic compounds by GC-MS is given in EPA Method 624.

VII. References:

Federal Register, December 3, 1979, pp 69532-69539, "Purgeables -  
Method 624."

USATHAMA Method 2J.

TABLE 3. INTERNAL STANDARD TO BE USED FOR  
COMPOUND QUANTIFICATION

<u>Compound</u>	<u>Internal Standard</u>
Benzene	2-bromo-1-chloropropane
Bromomethane	bromochloromethane
Chlorobenzene	1,4-dichlorobutane
1,2-Dichloroethane	bromochloromethane
Trans-1,2-Dichloroethene	bromochloromethane
1,1,2,2-tetrachloroethane	1,4-dichlorobutane
1,1,1-trichloroethane	bromochloromethane
Trichloroethene	2-bromo-1-chloropropane

F-3.0 IDENTIFICATION AND DETERMINATION OF SEMIVOLATILE ORGANIC  
COMPOUNDS IN WATER USING COMBINED FUSED SILICA CAPILLARY  
GAS CHROMATOGRAPHY AND MASS SPECTROMETRY

- I. Application: This method is used for the qualitative and semiquantitative determination of semivolatile organic compounds in water. Semivolatile organic compounds are defined as those organic compounds which can be extracted and concentrated by conventional solvent extraction techniques and pass through a fused silica capillary gas chromatographic column. Acidic, basic, and neutral compounds are determined in a single chromatograph run, by extracting the sample at both high and low pH and combining extracts.

This method is based on EPA Method 625 (see reference section), and the user should consult this method to establish its range of applicability. The use of a fused silica capillary column is recommended because of the superior resolving power compared to packed columns, i.e., less chance for interference and a faster rate of analysis.

Compounds which have been used to validate this method are listed below. Application of this method for the semiquantitative determination of other compounds is dependent on the chemical nature of such compounds.

A. Tested Concentration Range - ( $\mu\text{g/L}$  water)

Hexachloroethane - 5 to 100  
Naphthalene - 0.5 to 10  
Nitrobenzene - 2.5 to 50  
3,5-Dinitroaniline - 5 to 100  
2-Amino-4,6-dinitrotoluene - 5 to 100  
Fluoranthene - 0.5 to 10  
3-Nitrotoluene - 2.5 to 50  
Diethylphthalate - 0.5 to 10  
Alpha-BHC - 5 to 100  
pp'-DDT - 0.5 to 10  
Dieldrin - 0.5 to 10  
Lindane (Gamma BHC) - 5 to 100  
Heptachlor - 2.5 to 50  
Aroclor-1016 (PCB) - 50 to 1000  
Aroclor-1262 (PCB) - 50 to 1000  
2,4-Dinitrophenol - 5 to 100  
2-Methyl-4,6-dinitrophenol - 5 to 100  
Pentachlorophenol - 5 to 100  
Phenol (D6) - 0.5 to 10

- B. Sensitivity - The integrated area response at the detection limit for the most intense fragment ion of each compound in the final sample extract (1.0 mL) for a 2  $\mu$ L injection at the lowest concentration tested is presented below:

<u>Most Intense Mass Spectral Compound</u>	<u>Fragment Ion</u>	<u>Integrated Ion Peak Area</u>	<u>Concentration <math>\mu</math>g/l Water</u>
Hexachloroethane	119	1540	10
Naphthalene	128	1910	1
Nitrobenzene	123	1780	5
3,5-Dinitroaniline	183	3650	10
2-Amino-4,6-dinitrotoluene	180	2730	10
Fluoranthene	202	3040	1
3-Nitrotoluene	91	2860	5
Diethylphthalate	149	1490	1
Alpha-BHC	219	2120	10
pp'-DDT	235	690	1
Dieldrin	79	340	1
Lindane (gamma-BHC)	219	1680	10
Heptachlor	272	1150	5
Aroclor-1016 (PCB)	256*	5827	100
Aroclor-1262 (PCB)	360**	2743	100
2,4-Dinitrophenol	184	880	10
2-Methyl-4,6-dinitrophenol	198	2260	10
Pentachlorophenol	266	2890	10
Phenol (D6)	99	360	2

\*Ion of trichlorobiphenyl. Other ions are also quantitated.

\*\*Ion of hexachlorobiphenyl. Other ions are also quantitated.

\*\*\*Highest concentration tested.

C. Detection Limit - ( $\mu$ g/L water)

Hexachloroethane - 8  
 Naphthalene - 2  
 Nitrobenzene - 5  
 3,5-Dinitroaniline - 20  
 2-Amino-4,6-dinitrotoluene - 20  
 Fluoranthene - 3  
 3-Nitrotoluene - 6  
 Diethylphthalate - 5  
 Alpha-BHC - 30  
 pp'-DDT - 2  
 Dieldrin - 4  
 Lindane (gamma-BHC) - 30  
 Heptachlor - 9  
 Aroclor-1016 - 300  
 Aroclor-1262 - 300  
 2,4-Dinitrophenol - 40  
 2-Methyl-4,6-dinitrophenol - 40  
 Pentachlorophenol - 40  
 Phenol (D6) - 6

- D. Interferences - Compounds which coelute and have similar mass spectra to the compounds of interest may interfere.
- E. Analysis Rate - Approximately 1.5 hours are required to extract and concentrate a sample for GC-MS analysis. The GC-MS analysis time is 1.2 hour. One sample extractor and one GC-MS operator can analyze 5 samples in an 8 hour day.

## II. Chemistry

### A. Alternate Nomenclature and Chemical Abstracts Registry Number

Hexachloroethane:	$C_2Cl_6$ Carbon Hexachloride; perchloroethane CAS RN 67-72-1
Naphthalene:	$C_{10}H_8$ Napthalin; Napthene CAS RN 91-20-3
Nitrobenzene:	$C_6H_5NO_2$ Nitrobenzol CAS RN 98-95-3
3,5-Dinitroaniline:	$C_6H_5N_3O_4$ 3,5-Dinitrobenzenamine CAS RN 618-87-1
2-Amino-4,6-dinitrotoluene:	$C_7H_7N_3O_4$ 2-methyl -3,5-Dinitrobenzenamine CAS RN 35572-78-2
Fluoranthene:	$C_{16}H_{10}$ 1,2-Benzacenaphthene CAS RN 206-44-0
3-Nitrotoluene:	$C_7H_7NO_2$ 1-methyl-3-nitro-benzene CAS RN 99-08-1
Diethylphthalate:	$C_{12}H_{14}O_4$ 1,2-Benzenedicarboxylic Acid diethyl ester CAS RN 84-66-2
Alpha-BHC:	$C_6H_6Cl_6$ 1,2,3,4,6- Hexachlorocyclohexane CAS RN 319-84-6
pp'-DDT:	$C_{14}H_9Cl_5$ 1,1,1-Trichloro-2,2-bis(p- chlorophenyl)ethane; a,a- bis(p-chlorophenyl)-b,b,b,- Trichloroethane; dichloro diphenyl-trichloroethane; chlorophenstane; dicophane; pentachlorin CNS RN 50-29-3

Dieldrin:	$C_{12}H_8Cl_6O$ 1,2,3,4,10,10-hexachloro- exo-6,7-epoxy 1,4,4a,5,6,7,8a- octahydro-1,4-endo-exo-5,8- dimethanonaphthalene CAS RN 60-57-1
Lindane:	$C_6H_6Cl_6$ 1,2,3,4,6-hexachlorocyclo- hexane; HCH; Viton; GAMMA-BHC CAS RN 58-89-9
Heptachlor:	$C_{10}H_5Cl_7$ 1,4,5,6,7,8,8-heptachloro- 3a,4,7,7a-tetrahydro-4,7- methanoindene CAS RN 76-48-8
PCB-1016:	$C_{12}H_7Cl_3$ AROCHLOR 1016; Polychlorinated biphenyl with 41.5% Cl CAS RN 12674-11-2
PCB-1262:	AROCHLOR 1262; Polychlorinated biphenyl with 62% Cl CAS RN 37324-23-5
2,4-Dinitrophenol:	$C_6H_4N_2O_5$ 4,6-Aldifen CAS RN 51-28-5
2-Methyl-4,6-dinitrophenol:	$C_7H_6N_2O_5$ 4,6-Dinitro-o-cresol CAS RN 534-52-1
Pentachlorophenol:	$C_6Cl_5HO$ Santophen 20 CAS RN 87-86-5
Phenol (D6):	$C_6D_6O$ Perdeuterophenol CAS RN 13127-88-3



B. Physical and Chemical Properties

<u>Compound</u>	<u>Melting Point °C</u>	<u>Boiling Point °C</u>	<u>Density</u>	<u>Volume/Weight</u>
Hexachloroethane	187	186 (777mm)	$d_4^{20}$ 2.091	4.78 $\mu$ L/10 mg
Naphthalene	80.22	210.8 (720 mm)	1.145	Solid
Nitrobenzene	5.7	210.8	$d_4^{20}$ 1.2037	8.31 $\mu$ L/mg
3,5-Dinitroaniline	139-140	--	--	Solid
2-Amino-4,6-dinitrotoluene	--	--	--	Solid
Fluoranthene	111	375	1.252	Solid
3-Nitrotoluene	15	232.6	$d_4^{20}$ 1.1571	8.64 $\mu$ L/10 mg
Diethylphthalate	--	296	$d_4^{14}$ 1.2321	8.12 $\mu$ L/10 mg
Alpha-BHC	158	--	--	Solid
pp'-DDT	109	--	--	Solid
Dieldrin	176	--	--	Solid
Lindane	113	--	--	Solid
Heptachlor	95-96	--	--	Solid
PCB-1016	--	--	--	--
PCB-1262	--	--	--	--
2,4-Dinitrophenol	115-116	--	--	Solid
2-Methyl-4,6-dinitrophenol	87.5	--	--	Solid
Pentachlorophenol	174	309-310	1.978	Solid
Phenol (D6)	38-40	180	--	Solid
Decafluorobiphenyl	68.5	206	--	Solid
Pentafluorophenol	34-36	143	--	Solid
4-Fluoroaniline	--	187	1.156	8.65 $\mu$ L/10 mg

Use caution in handling the compounds listed above. Potential toxic inhalation and skin absorption hazard exist.

C. Chemical Reactions - N/AIII. Apparatus:

A. Instrumentation - Hewlett Packard 5985 gas chromatograph-mass spectrometer, or equivalent, equipped with a fused silica capillary column direct coupled to the mass spectrometer source. The system must be equipped with an appropriate data system to allow acquisition of full mass range scans for the duration of the chromatographic run. The computer should be equipped with mass storage devices for saving the data from the GC-MS. There should be computer software available to allow searching of any GC-MS run for specific ions and plotting the ions with respect to time or scan number. The ability to integrate the area under any specific ion plot is essential for quantification.

B. Parameters

1. Column - 30 m x 0.32 mm Fused Silica DB-5 30W (1.0  $\mu$ m, bonded phase equivalent to SE-54 available from J&W Scientific)
2. Gas flow - Helium at a linear velocity of 50 cm/sec (at about 15 psi, head pressure on the column, Manifold pressure =  $0.9 \times 10^{-5}$  Torr)
3. Temperatures - Injector - 240°C  
                             Source - 200°C  
                             Oven - 70°C (hold 1 min); then 10°C/min to 300°C (hold 11 min); total run time (including cool down) is 45 min
4. Injection volume - 2.0  $\mu$ L (splitless)
5. Detector - mass spectrometer scanned from 35-450 amu at 800 AMU/seconds
6. Retention times - (minutes)
  - Hexachloroethane - 6.6
  - Naphthalene - 8.3
  - Nitrobenzene - 6.7
  - 3,5-Dinitroaniline - 18.0
  - 2-Amino-4,6-dinitrotoluene - 18.4
  - Fluoranthene - 19.4
  - 3-Nitrotoluene - 8.4
  - Diethylphthalate - 13.8
  - Alpha-BHC - 15.4 min.
  - pp'-DDT - 22.0
  - Dieldrin - 20.6
  - Lindane (gamma-BHC) - 16.1
  - Heptachlor - 17.7
  - Aroclor-1016 (PCB) - 14.3 to 19.4 min.
  - Aroclor-1262 (PCB) - 20.0 to 26.0 min.
  - 2,4-Dinitrophenol - 12.8
  - 2-Methyl-4,6-dinitrophenol - 14.2

Pentachlorophenol - 16.0  
Phenol (D6) - 4.8  
Decafluorobiphenyl (ISTD) - 6.9  
Pentafluorophenol (ISTD) - 4.8  
4-Fluoroaniline (ISTD) - 5.0

C. Hardware/Glassware

1. 1-liter screw-capped bottles with Teflon-lined caps
2. metal spatula
3. wrist-action shaker
4. disposable glass pipets
5. 4-inch diameter glass funnel
6. volumetric flasks, 10.0-mL, 25.0-mL, 50.0-mL, 250.0-mL, Class A
7. syringes, 1.0-mL and 100- $\mu$ L
8. 30-mesh sieve
9. Kuderna-Danish (K-D) concentrator apparatus 250-mL, with Snyder columns
10. heated water bath
11. 2-Liter separatory funnel

D. Chemicals

1. methylene chloride, distilled in glass
2. acetone, distilled in glass
3. SARMS or interim SARMS
4. decafluorotriphenylphosphine (DFTPP)
5. decafluorobiphenyl; pentafluorophenol; 4-fluoroaniline (internal standards)

E. Reagents - N/A

IV. Standards

A. Calibration Standard

1. Individual Stocks were prepared at concentrations so that subsequent standard dilutions would be rendered more convenient. All "dinitro" analytes were made up at 20 mg/mL in chloroform, except 2-amino-2,4-dinitrotoluene which was made up at 10 mg/mL. The Aroclors 1016 and 1262 were also made up at 20 mg/mL. Pentachlorophenol, heptachlor, and dieldrin were made up at 5 mg/mL in chloroform. All other analytes were made up at 2.5 mg/mL in chloroform.

Stocks were prepared by weighing a 10 mL volumetric flask, adding about 25, 50, or 200 mg of reference material (using a syringe, glass capillary, or stainless steel spatula as appropriate), reweighing, and diluting to the mark with chloroform. Each solution is mixed well and stored protected from light in a glass tube with Teflon lined screw cap at -15 to -20°C. (Refer to Table 4.)

Table 4. Individual Stocks

	<u>Amount Added (mg)</u>	<u>Stock Concentration (mg/mL)</u>
Hexachloroethane	25	2.5
Naphthalene	25	2.5
Nitrobenzene	25	2.5
3,5-Dinitroaniline	200	20
2-Amino-4,6-dinitrotoluene	100	10
Fluoranthene	25	2.5
3-Nitrotoluene	25	2.5
Diethylphthalate	25	2.5
Alpha BHC	25	2.5
pp'-DDT	25	2.5
Dieldrin	50	5
Lindane	25	2.5
Heptachlor	50	5
Aroclor 1016	200	20
Aroclor 1262	200	20
2,4-Dinitrophenol	200	20
2-Methyl-4,6-dinitrophenol	200	20
Pentachlorophenol	50	5
Phenol (D6)	25	2.5

2. Stock A - Prepare Stock A solution to reflect the concentration range of calibration standards of each analyte. Into a 10 mL volumetric flask add 500  $\mu$ L of each Aroclor solution; 400  $\mu$ L each of alpha-BHC, gamma-BHC and hexachloroethane solutions; 200  $\mu$ L each of nitrobenzene, 3-nitrotoluene, and pentachlorophenol solutions; 100  $\mu$ L each of 2-amino-4,6-dinitrotoluene, and heptachlor solutions; 50  $\mu$ L each of 2,4-dinitrophenol, 2-methyl-4,6-dinitrophenol and 3,5-dinitroaniline solutions; 20  $\mu$ L of dieldrin solution; and 40  $\mu$ L of the remaining stock solutions.

Dilute to the mark with chloroform. Mix well and store in a tube with a Teflon-lined screw cap. Store protected from the light at  $-15^{\circ}$  to  $-20^{\circ}\text{C}$ . This procedure gives a solution having a final concentration at ten times the calibration LOD standards. The concentrations are about 1000  $\mu\text{g/mL}$  for each of the Aroclors; 100  $\mu\text{g/mL}$  for all of the "dinitro" compounds, pentachlorophenol, the volatile base-neutrals such as hexachloroethane, alpha-BHC, and gamma-BHC; 50  $\mu\text{g/mL}$  of nitrobenzene, 3-nitrotoluene, and heptachlor; and 10  $\mu\text{g/mL}$  of the remaining analytes. (Refer to Table 5.)

3. Internal Standard Stock - prepare the internal standard (IS) stock solution by dissolving 10 mg each of decafluorobiphenyl, pentafluorophenol, and 4-fluoroaniline in 5.0 mL of chloroform as described in A.1. above. This procedure gives a solution 2 mg/mL in each internal standard with chemical properties similar to those of the analytes in the acid, basic, and neutral fractions of the extracts.
4. GC-MS Calibration Standards - prepare the GC-MS calibration standard solutions by adding the volumes of Stock A solution given below to 5-mL volumetric flasks and diluting to volume with chloroform.

<u>Solution</u>	<u>Volume Added</u>		<u>Relative LOD Levels</u>
		<u>Stock A</u>	
Standard A	5.0	mL	10X
Standard B	2.5	mL	5X
Standard C	1.0	mL	2X
Standard D	0.5	mL	1X
Standard E	0.25	mL	0.5X

Store the GC-MS calibration standard solutions protected from light at  $-20^{\circ}\text{C}$ .

5. GC Operation Check Solution - prepare the GC Operation Check Solution by weighing 10 mg of decafluorotriphenyl-

Table 5. Stock A

	Amount of	Stock A
	<u>Stock Added (mg)</u>	<u>Concentration (mg/mL)</u>
Hexachloroethane	400	100
Naphthalene	40	10
Nitrobenzene	200	50
3,5-Dinitroaniline	50	100
2-Amino-4,6-dinitrotoluene	100	100
Fluoranthene	40	10
3-Nitrotoluene	200	50
Diethylphthalate	40	10
Alpha BHC	400	100
pp'-DDT	40	10
Dieldrin	20	10
Lindane	400	100
Heptachlor	100	50
Aroclor 1016	500	1000
Aroclor 1262	500	1000
2,4-Dinitrophenol	50	100
2-Methyl-4,6-dinitrophenol	50	100
Pentachlorophenol	200	100
Phenol (D6)	40	10

phosphine (DFTPP) into a 10-mL volumetric flask. Dilute to volume with toluene to produce a solution having a DFTPP concentration of 1 mg/mL. Prepare a dilution of this solution by diluting 250  $\mu$ L of it to a final volume of 10.0 mL with chloroform. The DFTPP concentration in this solution is 25 ng/ $\mu$ L.

- B. Control Spikes - A similar Stock A solution made up from methanolic standard stocks is used as the control spiking solution. An aliquot of Stock A is added to one liter of warm water. The aliquot should be sized such that the final concentration of each analyte in the spiked water sample is approximately 2 to 10 times the detection limit. Process the spiked sample by the preparation, extraction, and analysis procedure given below. Typical spike levels are given in the following table.

<u>Amount of Stock A in 1 Liter Water Sample (mL)</u>	<u>Spike Level (<math>\mu</math>g/L)</u>
0	0X
0.050	0.5X
0.100	1X
0.200	2X
0.500	5X
1.00	10X

- V. Procedure: The samples are extracted according to EPA Method 625. First the 1.0 L water sample is adjusted to pH 11 or greater with 6 N NaOH and extracted 3 times by shaking the sample with 60 mL aliquots of methylene chloride and allowing the phases to separate for at least 15 minutes before removing the organic phase. The extracted water sample is then adjusted to pH 2 or below with 6 N  $H_2SO_4$  and extracted 3 more times with 60 mL aliquots of methylene chloride. The acid extracts are then combined with the alkaline extracts prior to subsequent drying of the sample through a column of anhydrous sodium sulfate and sample concentration using Kuderna-Danish evaporation. Concentrate sample to a volume slightly less than 1.0 mL. Add about 50 mL chloroform and reconcentrate to about 1 mL. Adjust the final volume to 1.0 mL with chloroform. Spike extract with 10  $\mu$ L of the 2 mg/mL stock internal standard solution (equivalent to 20  $\mu$ g per liter in the original water sample) just prior to GC-MS analysis. Also add 10  $\mu$ L of the same internal standard solution to each mL of the calibration standards solutions. Prior to injecting any calibration standards or samples for GC-MS analysis, insure correct operation of the GC-MS system by injecting 2.0  $\mu$ L of the solution containing 25 ng/ $\mu$ L of decafluorotriphenylphosphine (DFTPP). The relative ion balances for proper operations are shown in Table 6.

TABLE 6. CORRECT ION ABUNDANCES OF DFTPP TO ENSURE PROPER TUNING AND SENSITIVITY OF THE MASS SPECTROMETER

<u>Mass</u>	<u>Abundance</u>
51	30-60% of mass 198
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	40-60% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	1% of mass 198
441	Less than mass 443
442	Greater than 40% of mass 198
443	17-23% of mass 442

The relative sensitivity of the instrument is determined by measuring the area of m/e 198 daily and recording the area counts on a control chart.

A hard copy of the DFTPP mass spectrum should be included with the reference and sample mass spectra acquired during the day's analyses. Inject the DFTPP standard at 70°C, hold for 1 minute, and then temperature program the GC column to 300°C at 10°C/min. Analyze the calibration standards beginning with the most concentrated, then samples. For each run determine the area under the peak of the characteristic fragment ion at the correct retention time for each compound of interest. Table 7 indicates the appropriate internal standard for use with each compound of interest.



Table 7. Internal Standard to be Used for  
Compound Quantification

<u>Compound</u>	<u>ISTD*</u>
Hexachloroethane	DFBP
Naphthalene	DFBP
Nitrobenzene	DFBP
3,5-Dinitroaniline	4FA
2-Amino-4,6-dinitrotoluene	4FA
Fluoranthene	DFBP
3-Nitrotoluene	DFBP
Diethylphthalate	DFBP
Alpha BHC	DFBP
pp'-DDT	DFBP
Dieldrin	DFBP
Lindane	DFBP
Heptachlor	DFBP
Aroclor 1016	DFBP
Aroclor 1262	DFBP
2,4-Dinitrophenol	PFP
2-Methyl-4,6-dinitrophenol	PFP
Pentachlorophenol	PFP
Phenol (D6)	PFP

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\*The Internal Standards (ISTD) are Decafluorobiphenyl (DFBP), Pentafluorophenol (PFP); and 4-fluoroaniline (4FA).

VI. Calculations: For each of the calibration standards determine the mass spectrometric response factor according to the following equation:

$$\text{Response Factor} = \text{RF} = \frac{(A_s) (C_{is})}{(A_{is}) (C_s)}$$

Where

$A_s$  is the integrated area of the characteristic ion for the pollutant standard.

$A_{is}$  is the integrated area of the characteristic ion of the internal standard (m/e 188).

$C_{is}$  is the amount ( $\mu\text{g}$ ) of the internal standard. Normally 20  $\mu\text{g}$ .

$C_s$  is the amount ( $\mu\text{g}$ ) of the pollutant standard.

The response factors should be relatively constant over the entire concentration range tested. Determine the mean, standard deviation, and percent relative standard deviation of the response factors. If the percent relative standard deviation of the response factor is no more than about 20%, use the mean response factor for computing compound concentrations. If the response factors vary significantly with concentration, prepare a calibration curve where the RF is plotted against the standard concentration. Once this calibration curve has been determined it should be verified daily by injecting at least one calibration standard. Calculate the daily concentration of each compound in the standard using the following equation and the appropriate response factor:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s) (C_{is})}{(A_{is}) (\text{RF})}$$

If the daily concentration differs more than 10% from the initial concentration, a new calibration curve must be determined by injecting new standards.

Use the following equation:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s) (C_{is})}{(A_{is}) (\text{RF})}$$

to calculate the concentration of each component in the sample using the mean response factor or appropriate response factor from the

calibration curve estimated from the relative area counts of the compound of interest relative to the internal standard. Additional criteria for quantitative determination of organic compounds by GC-MS are included in EPA Method 625.

VII. References:

Federal Register, December 3, 1979, pp 69540-69552, "Base/Neutrals, Acids and Pesticides - Method 625."

"Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry Systems," J.W. Eichelberger, L.E. Harris, and W.L. Budde, Anal Chem 47:995-1000 (1975).

## F.4.0 METALS IN WATER BY GRAPHITE FURNACE ATOMIC ABSORPTION

- I. Application: This method is applicable to the qualitative and semi-quantitative analysis of the following dissolved metals in natural water:

Arsenic (As)

Nickel (Ni)

Zinc (Zn)

A. Tested Concentration Range:

Arsenic (As) 2.5 to 50 µg/L

Nickel (Ni) 2.5 to 50 µg/L

Zinc (Zn) 0.5 to 10 µg/L

B. Sensitivity: (Absorbance Units)

Arsenic (As) 0.020 Abs.

Nickel (Ni) 0.038 Abs.

Zinc (Zn) 0.039 Abs.

C. Detection Limit (µg/L):

As - 4

Ni - 4

Zn - 3

D. Interferences: Interferences can be categorized as:

1. Spectral interferences which can be corrected by selection of an alternate wavelength, often accompanied by an increase in the detection limit.
2. Chemical interferences which can often be controlled by the addition of salts to the sample matrix.
3. Physical interferences which may be reduced by the utilization of standard addition techniques, and/or by the use of a deuterium arc background corrector.

- E. Analysis Rate: Approximately 30 samples can be analyzed for one analyte in a 3-hour period. This rate will vary depending on the specific analyte.

II. Chemistry:

A. Alternate Nomenclature and Chemical Abstracts Registry Number

B. Physical and Chemical Properties

<u>Metal</u>	<u>Melting Point (°C)</u>	<u>Boiling Point (°C)</u>
As	--	613 (sublimes)
Ni	1453	2732
Zn	420	907

III. Apparatus:A. Instrumentation:

Perkin-Elmer 5000 Atomic Absorption Spectrophotometer  
 Perkin-Elmer HGA-5000 Graphite Furnace  
 Perkin-Elmer AS-40 Auto Sampler, strip chart recorder and teletype

B. Parameters: Parameters indicated in Table 1 are suggested. Depending on various instrument conditions they can be varied.

C. Hardware/Glassware:

1. 2.5 mL autosampler cups
2. Pyrolytically coated graphite tubes
3. Volumetric flasks as needed
4. Volumetric pipets as needed
5. Micro pipets (with disposable tips)

D. Chemicals and Reagents:

1. 1000 ppm stock reference standards for applicable metals.
2. Deionized/distilled water.
3. Nickel Nitrate  $\text{Ni}(\text{NO}_3)_2$ , 1000 ppm: dissolve 0.796 g in deionized/distilled water and dilute to 100 mL.
4. Concentrated nitric acid, redistilled.

IV. Standards:

A. Calibration Standards: 1000  $\mu\text{g/mL}$  stock solutions of applicable metals are checked against SARMS, or 1000  $\mu\text{g/mL}$  SARM's can be used. These solutions are diluted as follows:

1. 1 mL of arsenic stock and 1 mL of nickel stock are placed in a 100 mL volumetric flask containing 1 mL redistilled  $\text{HNO}_3$ , and diluted to volume with deionized/distilled water. Final concentration is 10  $\mu\text{g/mL}$  for both metals, label standard A.
2. 10 mL of standard A is diluted to 100 mL in 1%  $\text{HNO}_3$ . Concentration 1  $\mu\text{g/mL}$  for both metals, label standard B.

3. Working standards for arsenic and nickel are prepared as follows:

<u>Volume of Standard (mL)</u>	<u>Dilution Volume (mL) in 1% HNO<sub>3</sub></u>	<u>Concentration (<math>\mu</math>g/L)</u>
0	50	0
0.10 of B	50	2
0.25 of B	50	5
0.50 of B	50	10
1.00 of B	50	20
0.50 of B	10	50
1.00 of B	10	100

4. 1 mL of zinc stock is placed in a 100 mL volumetric flask containing 1 mL of redistilled HNO<sub>3</sub>, and diluted to volume with deionized/distilled water. Final concentration is 10  $\mu$ g/mL for zinc, label standard C.
5. 2 mL of standard C is diluted to 100 mL in 1% HNO<sub>3</sub>. Final concentration is 0.2  $\mu$ g/mL, label standard D.
6. Working standards are prepared as follows:

<u>Volume of Standard (mL)</u>	<u>Dilution Volume (mL) in 1% HNO<sub>3</sub></u>	<u>Concentration (<math>\mu</math>g/L)</u>
0	50	0
0.10 of D	50	0.4
0.25 of D	50	1.0
0.50 of D	50	2.0
1.00 of D	50	4.0
0.50 of D	10	10.0
1.00 of D	10	20
0.25 of C	100	25

B. Control Spikes:

1. For arsenic and nickel, using 1000  $\mu$ g/mL stock solution, add 5 mL of each to a 100 mL volumetric flask containing 1 mL redistilled HNO<sub>3</sub> and dilute to volume. Dilute 1 mL of this solution to 100 mL in 1% HNO<sub>3</sub> to obtain the 500  $\mu$ g/L spiking solution.

Dilute the spiking solution with water in a 10 mL volumetric flask as indicated below. X is the estimated detection limit as indicated in Section I,C.

2. For zinc add 1 mL of 1000  $\mu$ g/mL stock to a 100 mL volumetric flask containing 1 mL of redistilled HNO<sub>3</sub> and dilute to volume. Dilute 1 mL of this solution to 100 mL in 1% HNO<sub>3</sub> to obtain the 100  $\mu$ g/L spiking solution.

Dilute the spiking solution with water in a 10 mL volumetric flask as indicated below. X is the estimated detection limit as indicated in Section I,C.

<u>Concentration (ug/L)</u>			<u>mL spike</u>	<u>Final Volume (mL)</u>
<u>As</u>	<u>Ni</u>	<u>Zn</u>		
0	0	0	0	10
2.5	2.5	0.5	0.05	10
5	5	1	0.10	10
10	10	2	0.20	10
25	25	5	0.50	10
50	50	10	1.00	10

- V. Procedure: Samples are placed in autosampler cups without prior treatments. Aliquots of each sample are then injected into the furnace and atomized according to the temperature program and instrument program. Values are printed on the teletype in absorbance units.

VI. Calculations:

- A. Standard Curve: Plot the instrument response for each analyte versus known concentration (in ug/L) and fit the best curve to the data.
- B. Concentration of Samples: Compare the instrument response for each analyte with the standard curve to determine the observed concentration (ug/L) for each samples.

VII. References:

1. "Analytical Methods for Furnace Atomic Absorption Spectroscopy," Perkin-Elmer Corporation, February 1980.
2. "Methods for Chemical Analysis of Water and Wastes," EPA, March 1980.

F-5.0 ANALYSIS OF METALS IN WATER BY  
INDUCTIVELY COUPLED PLASMA (ICAP) SPECTROSCOPY

- I. Application: The method is applicable to the semiquantitative analysis of the following metals in water:

Arsenic (As)	Lead (Pb)
Beryllium (Be)	Nickel (Ni)
Cadmium (Cd)	Silver (Ag)
Chromium (Cr)	Zinc (Zn)
Copper (Cu)	

- A. Tested Concentration Range: ( $\mu\text{g/l}$ )

Arsenic (As) 30 to 600	Lead (Pb) 12 to 240
Beryllium (Be) 0.2 to 4	Nickel (Ni) 6 to 120
Cadmium (Cd) 3 to 60	Silver (Ag) 2.5 to 50
Chromium (Cr) 2.5 to 50	Zinc (Zn) 6 to 120
Copper (Cu) 3 to 60	

- B. Sensitivity: (intensity in millivolts)

Arsenic (As) 8.9	Lead (Pb) 12.5
Beryllium (Be) 7.4	Nickel (Ni) 15.7
Cadmium (Cd) 13.9	Silver (Ag) 9.6
Chromium (Cr) 1.9	Zinc (Zn) 13.6
Copper (Cu) 9.1	

- C. Detection Limit ( $\mu\text{g/l}$  - estimated): (Method includes a 10X concentration factor).

<u>Calculated</u>	<u>Revised</u>	<u>Calculated</u>	<u>Revised</u>
As - 30	40	Pb - 20	30
Be - 0.4	0.4	Ni - 6	20
Cd - 6	6	Ag - 8	8
Cr - 4	20	Zn - 6	6
Cu - 6	6		

- D. Interferences: Concentrations of iron greater than 100,000  $\mu\text{g/l}$  are known to interfere with Cr and Pb, causing low results for Cr and high results for Pb. Iron concentrations of this magnitude are not anticipated in water samples.

- E. Analysis Rate: One analyst can prepare 40 samples in 8 hours. One analyst can analyze 10 samples per hour after instrument calibration.

II. Chemistry:

- A. Alternate Nomenclature and Chemical Abstract Number: None applicable.



B. Physical and Chemical Properties:

<u>Element</u>	<u>Molecular Weight</u>	<u>Melting Point (°C)</u>	<u>Boiling Point (°C)</u>
As	74.9	--	613 (sub)
Be	9.0	1278±5	2970
Cd	112.4	321	765
Cr	52.0	1857±20	2672
Cu	63.5	1083	2567
Pb	207.2	327	1740
Ni	58.7	1453	2732
Ag	107.9	962	2212
Zn	65.4	420	907

C. Chemical Reactions: None applicable.III. Apparatus:A. Instrumentation: Applied Research Laboratories, ICP quantometer, 38 fixed channels, PDP 1105 computer, Decwriter terminal, modified Gilson autosampler.B. Parameters:

1. Incident power: 1.6 KW
2. Reflected power: 0 W
3. Coolant flow: 11-12 l/min
4. Sample flow: 2-3 mL/min
5. Observation height: peaked
6. Analysis wavelength (nm):

As - 193.77	Pb - 220.35
Be - 234.86	Ni - 231.6
Cd - 226.5	Ag - 328.07
Cr - 267.72	Zn - 202.55
Cu - 324.75	

C. Hardware/Glassware:

1. Volumetric flasks: 25 mL, 1000 mL, others as needed.
2. Beakers: 500 mL Phillips, others as necessary for cleaning glassware.
3. Graduated cylinders: 250 mL
4. Pipets: 3 mL pipet head and flask, volumetric pipets as needed.

5. Watch glasses: 50 mm diameter
6. Hot plate
7. Wash bottle: 500 mL

D. Chemicals:

1. Nitric acid, redistilled
2. Nitric acid, reagent grade
3. Water, distilled/deionized (18 megohm/cm)
4. High purity metals and salts as indicated in Table 8.
5. SARMS for metals.

- E. Reagents: Nitric acid - reagent grade diluted 1:1 with laboratory water (for glassware washing).

IV. Standards:

- A. Calibration Standards: Prepared from high purity metals or high purity salts as indicated in Table 9. Stock solutions are prepared from or checked against SARMS.

- B. Control Spikes: See Table 10.

1. Place 1 mL redistilled  $\text{HNO}_3$  into each of two 1000 mL volumetric flasks. Add approximately 900 mL deionized/distilled water.
2. Add the following volumes (mL) of the SARM (1000 ppm) for each metal to the flasks.

<u>Flask I</u>	<u>Flask II</u>
As - 15.0	Cr - 1.25
Be - 0.10	Cu - 1.50
Cd - 1.50	Pb - 6.00
	Ni - 3.00
	Ag - 1.25
	Zn - 3.00

Then dilute to volume.

3. Spike the volumes indicated in Table 10 from each of the flasks into 250 mL of water and then prepare for analysis the same as the sample.

Table 8.  
Stock Calibration Standards

<u>Group</u>	<u>Elements</u>	<u>Prepared From [Amount Used (mg/100 ml stock)]</u>	<u>Dissolved In</u>	<u>Stock Conc.</u>
1	Cu	Metal (1000)	HNO <sub>3</sub>	1%
	Cd	Metal (1000)	HNO <sub>3</sub>	1%
	Ag	AgNO <sub>3</sub> (1575)	HNO <sub>3</sub>	1%
	Be	Metal (100)	HNO <sub>3</sub>	1000 ppm
2	Zn	ZnO (1245)	HCl	1%
3	Cr	Metal (1000)	HNO <sub>3</sub>	1%
	Ni	NiO (1273)	HNO <sub>3</sub>	1%
	As	Metal (1000)	HNO <sub>3</sub>	1%
4	Pb	Metal (1000)	HNO <sub>3</sub>	1%

Table 9.  
Working Calibration Standards  
(concentrations in µg/mL)

Group	Elements	mL Stock	Standard A Conc. Diluted to 100 mL	Standard B (A Diluted 5/50)	Standard C (B Diluted 5/50)	Standard D (C Diluted 10/25)	Standard E (D Diluted 5/50)	Standard F (E Diluted 20/100)
1	Cu	3	300	30	3	1.2	.3	.06
	Cd	1	100	10	1	.4	.1	.02
	Ag	2	200	20	2	.8	.2	.04
	Be	2	20	2	0.2	.08	.02	.004
2	Zn	2	200	(A Diluted 10/25)	(A Diluted 5/50)	(C Diluted 5/50)	(D Diluted 5/50)	(E Diluted 20/100)
				80	20	2	.2	.04
3	Cr Ni As	2	200	(A Diluted 5/50)	(B Diluted 5/50)	(C Diluted 10/25)	(C Diluted 10/100)	
		2	200	20	2	.8	.2	
		4	400	40	4	1.6	.4	
		3	300	30	3	1.2	.3	

Table 10.

Control Spikes

Conc. of Spiking Solution ( $\mu\text{g/ml}$ ) -->	As	Be	Cd	Cr	Cu	Pb	Ni	Ag	Zn
15	0.1	1.5	1.25	1.5	6	3	1.25	3	
ml Spiking Solution*									
Spikes	<u>Added to 250 ml</u>								
	<u>Conc. of Spikes (<math>\mu\text{g/l}</math>)</u>								
Blank	0	0	0	0	0	0	0	0	0
0.5X	0.5	30	0.2	3	2.5	3	12	6	2.5
1X	1	60	0.4	6	5	6	24	12	5
2X	2	120	0.8	12	10	12	48	24	10
5X	5	300	2.0	30	25	30	120	60	25
10X	10	600	4.0	60	50	60	240	120	50
									120

\*Volume Flask I and Flask II combined in the same 250 ml flask.

- V. Procedure: Clean all glassware by soaking in hot 1:1  $\text{HNO}_3$  and then rinsing in copious amounts of distilled and deionized/distilled water. Transfer 200 mL of the well mixed, acid preserved, sample to a Phillips beaker. Add 3 mL of concentrated  $\text{HNO}_3$  (redistilled). Place the beaker on the hot plate and evaporate to approximately 5 mL (add an additional 50 mL of sample to the beaker during evaporation so that the final sample volume used is 250 mL). Do not allow the sample to boil or to evaporate to dryness. Cool the beaker and add an additional 3 mL of concentrated  $\text{HNO}_3$  (redistilled). Cover the beaker with a watch glass and heat so that a gentle reflux occurs. Continue heating until digestion is complete. Cool the beaker, rinse the watch glass and the walls of the beaker with 2-3 mL of distilled/deionized water. Quantitatively transfer the digested sample to a 25 mL volumetric flask containing 3 mL of concentrated  $\text{HNO}_3$  (redistilled) and dilute to volume.
- VI. Calculations:
- A. Standard Curve: Plot the instrument response for each analyte versus known concentration (in  $\mu\text{g/l}$ ) and fit the best curve to the data.
- B. Concentration of Samples: Compare the instrument response for each analyte with the standard curve to determine the observed concentration ( $\mu\text{g/l}$ ) for each sample. (The calculation of a standard curve, and the comparison of sample data with this curve, is performed by the instrument's computer.) Subtract the concentration found in the instrument blank from each observed concentration and divide by 10 to determine the concentration of the original sample ( $\mu\text{g/l}$ ).
- VII. Reference: USATHAMA Method 3T, revised 23 January 1981.

F-6.0 IDENTIFICATION AND DETERMINATION OF EXPLOSIVES AND RELATED  
MATERIALS IN WATER USING HIGH PERFORMANCE LIQUID  
CHROMATOGRAPHY (HPLC)

- I. Application: This method is designed primarily as a semi-quantitative HPLC screening method for explosives and related materials in water. The method has been used to semiquantitatively determine the following compounds in water samples:

2,4-Dinitrotoluene	- 2,4-DNT
2,6-Dinitrotoluene	- 2,6-DNT
2,4,6-Trinitrotoluene	- 2,4,6-TNT
Tetryl	- Tetryl
RDX	- RDX

- A. Tested Concentration Range: ( $\mu\text{g/l}$  water)

2,4-DNT	- 0.6 to 12
2,6-DNT	- 0.6 to 12
2,4,6-TNT	- 0.5 to 10
Tetryl	- 0.5 to 10
RDX	- 0.5 to 10

- B. Sensitivity: Response (integrator peak height) at the detection limit.

2,4-DNT	- 2000
2,6-DNT	- 2600
2,4,6-TNT	- 1200
Tetryl	- 2000
RDX	- 5600

- C. Detection Limit: ( $\mu\text{g/l}$  water)

2,4-DNT	- 3
2,6-DNT	- 4
2,4,6-TNT	- 1
Tetryl	- 2
RDX	- 2

- D. Interferences: Compounds which coelute with the compounds of interest, and absorb 254 nm UV radiation will interfere.

- E. Analysis Rate: One analyst can analyze 8 samples in an 8 hour day.

## II. Chemistry

### A. Alternate Nomenclature and Chemical Abstracts Registry Number:

2,4-Dinitrotoluene: CAS RN 121-14-2

2,6-Dinitrotoluene: CAS RN 606-20-2.

2,4,6-Trinitrotoluene: svm-trinitrotoluene; 1-methyl-2,4,6-trinitrobenzene; trotyl; Tolit; Trilit: CAS RN 118-96-7.

Tetryl: nitramine; N-methyl - N, 2,4,6-Tetranitrobenzinamine; N-methyl - N-2,4,6-tetranitroaniline; picrylmethylnitramine; picrylnitromethylamine; Tetralite. CAS RN 479-45-8.

RDX: Hexahydro-1,3,5-trinitro-1,3,5-triazine;  $T_4$ ; cyclonite; Hexogen; cyclotrimethylenetrinitramine. CAS RN 121-82-4.

### B. Physical and Chemical Properties:

2,4-DNT:  $C_7H_6N_2O_4$ ; m.p. =  $71^{\circ}C$ ; b.p. =  $300^{\circ}C$  (decomposes)

2,6-DNT:  $C_7H_6N_2O_4$ ; m.p. =  $66^{\circ}C$ .

2,4,6-TNT:  $C_7H_5N_3O_6$ ; m.p. =  $82^{\circ}C$ ; b.p. =  $240^{\circ}C$  (explodes)

Tetryl:  $C_7H_5N_5O_8$ ; m.p. =  $131^{\circ}C$ ; b.p. =  $187^{\circ}C$  (explodes)

RDX:  $C_3H_6N_6O_6$ ; m.p. =  $205^{\circ}C$ .

Hazards: Several of these compounds are explosives. Use caution in handling. Potential toxic inhalation and skin absorption hazards exist.

### C. Chemical Reactions: N/A

## III. Apparatus

A. Instrumentation: Spectra-Physics SP-8700 ternary solvent delivery system, Spectra-Physics SP-4100 computing integrator, Perkin Elmer Model LC-75 UV-Vis variable wavelength detector with Model LC-75 autocontrol, Waters WISP Model 710B auto-sampler.

### B. Parameters

1. Column - Supelco RP-2, 4.6 mm x 250 mm, 5 micron particle size
2. Solvent program - 40% methanol/60% water, isocratic elution
3. Flow - 1 ml/min.
4. Detector - 254 nm @ 0.01 AUFS
5. Injection volume - 100  $\mu$ l



## 6. Retention times -

2,4-DNT	- 13.35 min
2,6-DNT	- 14.66 min
2,4,6-TNT	- 9.85 min
Tetryl	- 11.03 min
RDX	- 7.59 min

C. Hardware/Glassware:

1. 2-l bottles with polyseal caps.
2. 2-dram vials with polyseal caps.
3. Volumetric flasks, 5-ml, 10-ml, 25-ml, 50-ml, 100-ml, 250-ml.
4. Pipets, 1-ml, 2-ml, 5-ml, 10-ml.
5. Millipore all glass filter apparatus
6. Millipore filters, 0.45  $\mu$  type HA, 47 mm
7. Syringes, 1-ml and 100- $\mu$ l

D. Chemicals:

1. Methanol, distilled in glass.
2. Water, Milli-Q or equivalent.
3. SARMS or interim SARMS for standard solutions.

E. Reagents: N/AIV. Standards:A. Calibration standards:

1. Stock A - weigh the following amounts of each SARM into separate 25-ml volumetric flasks.

<u>Compound</u>	<u>Amount</u>	<u>Final Concentration</u>
2,4-DNT	30 mg	1.2 mg/ml
2,6-DNT	30 mg	1.2 mg/ml
2,4,6-TNT	25 mg	1 mg/ml
Tetryl	25 mg	1 mg/ml
RDX	25 mg	1 mg/ml

Dissolve in methanol, dilute to volume, and mix well. Stock standards are stored protected from light at  $-20^{\circ}\text{C}$ .

2. Stock B - Add 1 ml of each stock A to a 10-ml volumetric flask. Dilute to volume with methanol and mix well. The resulting solution is stored protected from light at  $-20^{\circ}\text{C}$ . The mixed stock has a final concentration of 100  $\mu\text{g/ml}$  for 2,4,6-TNT, tetryl, and RDX, and 120  $\mu\text{g/ml}$  for 2,4-DNT and 2,6-DNT.

3. Working Standards: Prepare working standards by making dilutions in a volumetric flask with 40% methanol/60% water of Stock B as follows:

<u>Solution</u>	<u>Amount of Mixed Stock B</u>	<u>Dilution</u>	<u>Final Concentration</u>	
			<u>TNT, RDX, Tetryl</u>	<u>2,4-DNT and 2,6-DNT</u>
Standard A	0 $\mu$ l	5 ml	0 $\mu$ g/ml	0 $\mu$ g/ml
Standard B	1.5 $\mu$ l	10 ml	0.015 $\mu$ g/ml	0.018 $\mu$ g/ml
Standard C	4 $\mu$ l	10 ml	0.04 $\mu$ g/ml	0.048 $\mu$ g/ml
Standard D	10 $\mu$ l	10 ml	0.1 $\mu$ g/ml	0.12 $\mu$ g/ml
Standard E	10 $\mu$ l	5 ml	0.2 $\mu$ g/ml	0.24 $\mu$ g/ml
Standard F	25 $\mu$ l	5 ml	0.5 $\mu$ g/ml	0.6 $\mu$ g/ml
Standard G	50 $\mu$ l	5 ml	1.0 $\mu$ g/ml	1.2 $\mu$ g/ml

Standards A through G are stored protected from light at  $-20^{\circ}\text{C}$ .

- B. Control Spikes: The control spiking solution is Stock B solution. Pipet a known amount of spiking solution into a bottle containing a 500-ml water sample aliquot to be spiked. The amount pipetted should give a spike level having a concentration around the method detection limit. Perform calibration and analyze the spiked samples with the procedure given below. Unspiked water samples are also analyzed as controls. Suggested spike levels are given in the following table.

<u>Amounts of Stock B in 500 ml of Water (<math>\mu</math>l)</u>	<u>Final Concentration (<math>\mu</math>g/l)</u>	
	<u>TNT, RDX, Tetryl</u>	<u>2,4-DNT and 2,6-DNT</u>
0	0	0
2.5	0.5	0.6
5	1	1.2
10	2	2.4
25	5	6
50	10	12

#### V. Procedure:

- A. Sample Preparation: Place 500 ml of the water sample in a one liter separatory funnel. Extract with 100 ml of methylene chloride, shaking vigorously for at least three minutes, and allow the layers to separate. Drain the methylene chloride into a 500 ml Kuderna-Danish receiver. Repeat this procedure with two 50 ml portions of methylene chloride, adding each portion to the Kuderna-Danish receiver. Attach a 3 ball Snyder column to the receiver and concentrate in a water bath at  $70^{\circ}\text{C}$ . When the volume is less than 10 ml, quantitatively transfer the sample to a 10-ml concentrator tube and re-concentrate under a stream of dried nitrogen in a water bath at  $30^{\circ}\text{C}$ . When the volume is approximately one ml, add 2 ml of methanol and re-concentrate to 1 ml; repeat two more times. Bring the final volume to exactly 2 ml with methanol, then add exactly 3 ml of HPLC water. Sample may then be injected directly into liquid chromatograph.

- B. Calibration: Inject 100  $\mu$ l of each standard onto LC column for calibration. Record retention time, concentration, and peak area for each component.
- C. Sample Analysis: Use the calibration standard solution results for each compound to construct a plot of peak height versus component concentration in  $\mu$ g/l. Determine the linear regression analysis equation for these data. Determine the amount of analyte in the sample from the regression equation.

VI. Calculations:

Convert calibration standard concentrations ( $\mu$ g/ml) to  $\mu$ g/L using the following formula:

$$(\mu\text{g/ml}) \times 10 = \mu\text{g/L}$$

Use these calculated values to construct the calibration curve.

VII. References:

USATHAMA Method 3S, No. 5, Revised January 23, 1981.

F-7.0 ANALYSIS OF SODIUM IN WATER BY  
ATOMIC ABSORPTION SPECTROSCOPY

I. Application: This method is applicable to the qualitative and semi-quantitative analysis of sodium in water.

A. Tested Concentration Range: 0.5 to 10 mg/L.

B. Sensitivity: 0.025 Abs at 1 mg/L

C. Detection Limit: 1 mg/L

D. Interferences: None

E. Analysis Rate: One analyst can analyze 20 samples per hour after instrument calibration.

II. Chemistry

A. Alternate Nomenclature and Chemical Abstract Number: None applicable.

B. Physical and Chemical Properties

Molecular Weight - 23.0

Melting Point - 98°C

Boiling Point - 892°C

C. Chemical Reactions: None applicable.

III. Apparatus

A. Instrumentation: Instrumentation Laboratories 751 atomic absorption/emission spectrophotometer (UBTL designated "B" instrument); Sodium hollow cathode lamp.

B. Parameters

1. Lamp Current: 8

2. Bandpass: 1

3. Wavelength: 589.6 nm

4. Flame: Air/acetylene

5. Burner head turned 90°C

6. Other parameters as specified by instrument manufacturer.

C. Hardware/Glassware

1. Volumetric Flasks: 100 mL, 200 mL, others as needed

2. Volumetric pipetes: as needed

#### D. Chemicals

1. Nitric acid, redistilled
2. Water, deionized/distilled (18 megohm/cm)
3. 1000 ppm SARM for sodium

#### IV. Standards

- A. Calibration Standards: Place 10 mL of the SARM into a 100 mL volumetric flask containing 1 mL of redistilled  $\text{HNO}_3$  and dilute to volume; label "A." Prepare working standards as follows:

<u>mL Stock A</u>	<u>Dilution Volume (mL)</u>	<u>Concentration of Standard (mg/L)</u>
0	100	0
1	200	.5
1	100	1
2	100	2
5	100	5
10	100	10
25	100	25

- B. Control Spikes: Spike 100 mL of water with the 1000 ppm SARM as indicated below:

<u>mL SARM</u>	<u>Concentration of Spike mg/L</u>
0	0
.050	0.5
.100	1
.200	2
.500	5
1.000	10

- V. Procedure: The untreated sample is aspirated directly into the flame of the atomic absorption spectrophotometer and the absorbance recorded.

#### VI. Calculations

- A. Standard Curve: Plot the absorbance reading for each standard versus known concentration (mg/L) and fit the best curve to the data.
- B. Concentration of Samples: Compare the instrument response for each sample and determine the observed concentration.

- VII. Reference: "Methods for Chemical Analysis of Water and Wastes," EPA, March 1980.

## F-8.0 DETERMINATION OF TOTAL CYANIDE IN WATER

I. Application: This method is applicable to the determination of total cyanide in drinking water, surface and saline waters, domestic and industrial wastes.

- A. Tested Concentration Range: 5 µg/L to 100 µg/L
- B. Sensitivity: 0.070 Absorbance Units at 10 µg/liter
- C. Detection Limit: 5 µg/L
- D. Interferences: Interferences are eliminated or reduced by using the distillation procedure.

Sulfides adversely affect the procedure. If a drop of the distillate on lead acetate test paper indicates the presence of sulfides, treat 25 ml more of the sample than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide.

Fatty acids will distill and form soaps under the alkaline titration conditions, making the end point almost impossible to detect. If that occurs, the following procedure may be used.

Acidify the sample with acetic acid (1+9) to pH 6.0 to 7.0.

Caution: This operation must be performed in the hood and the sample left there until it can be made alkaline again after the extraction has been performed.

Extract with iso-octane, hexane, or chloroform (preference in order named) with a solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the fatty acids below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with NaOH solution.

- E. Analysis Rate: One analyst can analyze six samples in an 8 hour day.

## II. Chemistry:

- A. Alternate Nomenclature and Chemical Abstracts Registry Number: Total cyanide is defined as cyanide ion ( $\text{CN}^-$ ) and complex cyanides which are converted to hydrogen cyanide (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.
- B. Physical and Chemical Properties: Hydrogen cyanide, which is released at the low pH encountered in this method, is an acute poison. Operations so designated must be performed in the hood.
- C. Chemical Reactions: The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined colorimetrically. In the colorimetric measurement the cyanide is converted to cyanogen chloride,  $\text{CNCl}$ , by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 578 nm.

## III. Apparatus

- A. Instrumentation: A Coleman 54B spectrophotometer with a 2.5 cm cuvette will be used to determine the absorbance.
- B. Parameters: The spectrophotometer will be zeroed at 578 nm against zero standard.
- C. Hardware/Glassware: A reflux distillation apparatus as shown in Figure 1 is used with a 1 liter boiling flask.
- D. Chemicals:
  1. Sodium hydroxide solution, 1.25N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water.
  2. Cadmium carbonate: powdered.
  3. Ascorbic acid: crystals.
  4. Dilute sodium hydroxide solution, 0.25N: Dilute 200 ml of sodium hydroxide solution (D.1.) to 1000 ml with distilled water.
  5. Sulfuric acid: (1+1)  $\text{H}_2\text{SO}_4$
  6. Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$  in 1 liter of distilled water. Refrigerate this solution.
  7. Potassium cyanide: reagent grade or SARM
  8. Silver nitrate: reagent grade or SARM
  9. Rhodanine indicator: Dissolve 20 mg of p-dimethyl-amino-benzalrhodanine in 100 ml of acetone.
  10. Chloramine T solution: Dissolve 1.0 g of white, water soluble Chloramine T in 100 ml of distilled water and refrigerate until ready to use. Prepare fresh weekly.

11. Pyridine-Barbituric Acid Reagent: Place 15 g of barbituric acid in a 250 ml volumetric flask and add just enough distilled water to wash the side of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15 ml of HCl (sp gr 1.19) mix, and cool to room temperature. Dilute to 250 ml with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.
12. Magnesium chloride solution: Weigh 510 g of  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  into a 1000 ml flask, dissolve and dilute to 1 liter with distilled water.
13. Potassium chromate indicator solution: Dissolve 50 g  $\text{K}_2\text{CrO}_4$  in a little distilled water. Add  $\text{AgNO}_3$  solution until a definite red precipitate is formed. Let stand 12 hr, filter, and dilute to 1 L with distilled water.

#### IV. Standards:

##### A. Calibration Standards:

1. Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in one liter of distilled water.
2. Stock silver nitrate solution (approximately 0.0192N): Weigh out 3.27 g dried  $\text{AgNO}_3$  and dissolve in one liter of distilled water.
3. Primary standard sodium chloride solution: Dry NaCl at  $140^\circ\text{C}$  for one hour. Cool, weigh out 1.1222 g of NaCl and dissolve in one liter of distilled water. The final solution is 0.0192 N in NaCl.
4. Standardization of stock silver nitrate solution: Pipet 5 ml of primary standard sodium chloride solution into a 250 ml flask. Dilute to approximately 100 ml with distilled water. Adjust the pH to between 7 and 10 with 0.05 N NaOH. Add 1 ml of potassium chromate indicator. Titrate with stock silver nitrate solution to the pinkish yellow endpoint. A blank of 0.2 to 0.3 ml is usual. Calculate the normality of the  $\text{AgNO}_3$  solution.
5. Standardization of stock cyanide solution: Pipet 5 ml of stock cyanide solution into a 250 ml flask. Dilute to approximately 100 ml with distilled water. Prepare a blank consisting of 3.6 ml of 0.05 N NaOH solution diluted to 100 ml. Add 0.5 ml rhodanine indicator solution. Titrate with standardized silver nitrate solution to a salmon colored endpoint. Correct for the blank value and calculate mg per liter CN. Note that 0.0192 N  $\text{AgNO}_3$  is equivalent to 1 mg CN per ml.



6. Preparation of intermediate cyanide solution (10 mg per liter): Pipet 10 ml of standardized stock cyanide solution (1 mg/ml) into a one liter volumetric flask and dilute to the mark with 0.05 N NaOH solution.
7. Preparation of standard cyanide working solution (1 mg/liter): Pipet 100 ml of intermediate cyanide solution (10 mg/liter) into a 1000 ml volumetric flask and dilute to the mark with 0.05 N NaOH solution.
8. Prepare a series of standards by pipeting suitable volumes of standard cyanide working solution (1 mg/liter) into 50 ml volumetric flasks. Each standard is brought to 20 ml with 0.05 N NaOH as described in the following table.

ML of Standard Cyanide Solution (1 mg/liter)	ML of 0.05 N NaOH Solution	µg Cyanide
0	20	0
0.1	19.9	0.1
0.2	19.8	0.2
0.4	19.6	0.4
1.0	19.0	1.0
2.0	18.0	2.0

The standards are prepared for colorimetric analysis in accordance with section V.2. below.

- B. Control Spikes: The spiking solution is the Standard Cyanide Working Solution (1 mg/liter). Prepare a series of control spikes by pipeting suitable volumes of standard solution into one liter volumetric flasks. To each standard add 200 ml of 0.25 N sodium hydroxide solution and dilute to one liter with distilled water. Prepare as follows:

ML of Standard Working Cyanide Solution (1 mg/liter)	Final Concentration µg/liter Cyanide
0	Blank
5	5
10	10
20	20
50	50
100	100

#### V. Procedure:

1. Place 500 ml of sample, or an aliquot diluted to 500 ml in the 1 liter boiling flask. Add 50 ml of 0.25 N sodium hydroxide to the absorbing tube and dilute if necessary with distilled water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train.

Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

Caution: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

Slowly add 50 ml 1+1 sulfuric acid through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 min. Pour 20 ml of magnesium chloride solution into the air inlet and wash down with a stream of water.

Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

Drain the solution from the absorber into a 250 ml volumetric flask and bring up to volume with distilled water washings from the absorber tube.

2. Withdraw 20 ml or less of the solution from the flask and transfer to a 50 ml volumetric flask. If less than 20 ml is taken, dilute to 20 ml with 0.05 N sodium hydroxide solution. Add 4 ml of 1M Sodium phosphate solution and mix. Add 2 ml of chloramine T and mix. Immediately, add 5 ml of pyridine - barbituric acid solution and mix. Dilute to mark with distilled water and mix again. Allow 8 minutes for color development then read absorbance at 578 nm in a 2.5 cm cell within 15 minutes.

VI. Calculations: Calculate the cyanide in  $\mu\text{g/L}$  in the original sample as follows:

$$\text{CN, } \mu\text{g/L} = \frac{A \times B \times 1,000}{C \times D}$$

where:

A =  $\mu\text{g}$  CN read from standard curve (50 ml final volume)

B = total volume of absorbing solution from the distillation, 250 ml

C = volume of original sample used in the distillation, 500 ml

D = aliquot of absorbing solution used in colorimetric test, 20 ml

VII. References:

- A. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979, Method 335.2
- B. Standard Methods for the Examination of Water & Wastewater, 15th Edition, 1980 (APHA-AWWA-WPCF).

## F-9.0 DETERMINATION OF MERCURY IN WATER (QUALITATIVELY)

I. Application: This method is applicable to the determination of mercury in water qualitatively.

A. Tested Concentration Range: 0.2  $\mu\text{g/l}$  to 1.0  $\mu\text{g/l}$ .

B. Sensitivity: 59 mm at 0.2  $\mu\text{g/l}$ .

C. Detection Limit: 0.2  $\mu\text{g/l}$ .

D. Interferences: Using the combination of digestion reagents listed in this method, interferences are eliminated.

E. Analysis Rate: One analyst can digest 36 samples in 5 hours and analyze 36 samples in an 8-hour period.

II. Chemistry:

Mercury

CAS RN 7439-97-6

Melting point:  $-39^{\circ}\text{C}$

Boiling point:  $357^{\circ}\text{C}$

III. Apparatus:

A. Instrumentation: Perkin Elmer Model 303 atomic absorption spectrophotometer equipped with a quartz window cell, peristaltic pump, bubbler, strip chart recorder.

B. Parameters:

1. Wavelength: 253.6 nm
2. Slit: 5
3. Hollow cathode mercury lamp: current 6 ma
4. Chart range: 10 mV

C. Hardware/Glassware:

1. 300 ml BOD bottles
2. Microliter pipettes with disposable tips
3. Hot water bath
4. Reagent dispensers
5. Graduated cylinders as needed
6. Volumetric flasks as needed

D. Chemicals:

1. Concentrated sulfuric acid reagent grade.
2. Concentrated nitric acid, redistilled.

3. Potassium permanganate, 5% solution; weigh 25 g  $\text{KMnO}_4$  into a 500 ml volumetric flask and dilute to volume with deionized/distilled water.
4. Potassium persulfate, 5% solution; weigh 25 g  $\text{K}_2\text{S}_2\text{O}_8$  into a 500 ml volumetric flask and dilute to volume with deionized/distilled water.
5. Hydroxylamine hydrochloride solution; weigh 20 g  $\text{NH}_2\text{OH}\cdot\text{HCl}$  into a 250 ml volumetric flask and dilute to volume with deionized/distilled water.
6. Hydrochloric acid, 1:1; add 150 ml concentrated HCl to 150 ml deionized/distilled water.
7. Stannous chloride solution; weigh 25 g  $\text{SnCl}_2$  into a 250 ml volumetric flask and dilute to volume with 1:1 HCl.

#### IV. Standards:

- A. Calibration Standards: USATHAMA 1 mg/ml Hg standard analytical reference material. Prepare working calibration standards fresh daily as follows:

1. Place 0.200 ml of 1 mg/ml standard into a 100 ml volumetric flask containing 1 ml redistilled nitric acid and dilute to volume with deionized/distilled water (Solution A, 2.0  $\mu\text{g}/\text{ml}$ ).
2. Place 5 ml of Solution A into a 50 ml volumetric flask containing 0.1 ml of redistilled nitric acid and dilute to volume with deionized/distilled water (Solution B, 0.20  $\mu\text{g}/\text{ml}$ ).
3. Place 100 ml deionized/distilled water into each of 14 BOD bottles.
4. Spike, into duplicate bottles, the following volumes of Solution B (0.20  $\mu\text{g}/\text{ml}$ ):

<u>Spike Volume (<math>\mu\text{l}</math>)</u>	<u><math>\mu\text{g}/\text{Bottle}</math></u>	<u><math>\mu\text{g}/\text{liter}</math></u>
0	0	0
50	0.0100	0.100
100	0.0200	0.200
200	0.0400	0.400
500	0.1000	1.00
1000	0.2000	2.00

5. Standards are then prepared and analyzed in the same manner as the sample.

B. Control Spikes: Control spikes are prepared in the same manner as the calibration standards.

V. Procedure: Transfer 100 ml, or an aliquot diluted to 100 ml, containing not more than 0.5 µg of mercury, to a 300 ml BOD bottle. Add 5 ml of sulfuric acid and 2.5 ml of concentrated nitric acid, mixing after each addition. Add 15 ml of potassium permanganate solution to each sample bottle. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 minutes. Add 8 ml of potassium persulfate to each bottle and heat for 2 hours in a water bath at 95°C. Cool and add 6 ml of hydroxylamine hydrochloride to reduce the excess permanganate. After a delay of at least 30 seconds, add 5 ml of stannous chloride and immediately attach the bottle to the aeration apparatus. Leave the bottle attached to the apparatus until no further increase in peak height is observed on the strip chart recorder.

VI. Calculation:

A. Standard Curve: Plot the peak height versus known concentration (in µg/bottle) and fit the best curve to the data.

B. Concentration of Samples: Compare the peak height with the standard curve to determine the observed concentration (in µg/bottle) for each sample. Use the following formula to compute µg/L:

$$\frac{[(\mu\text{g/bottle}) - \text{reagent blank}]}{\text{sample aliquot size in mL}} \times 1000 = \mu\text{g/L}$$

VII. Reference: EPA "Analytical Methods for the Analysis of Water and Wastes," March 1979, Method No. 245.1.

## P-10.0 DETERMINATION OF GROSS ALPHA AND GROSS BETA ACTIVITY IN WATER

- I. Application: This method describes the procedure for the determination of gross alpha and gross beta activity in waters. This activity is not indicative of any specific nuclide. However, it does provide an index to the level of gross activity of the samples semi-quantitatively.

A. Tested Concentration Range

Gross Alpha            1 pCi/l to 10,000 pCi/l in water

Gross Beta            1.5 pCi/l to 10,000 pCi/l in water

B. Sensitivity - Response-1 count per minute over background at the detection limit

Gross Alpha            2 pCi/l

Gross Beta            3 pCi/l

C. Detection Limit

Gross Alpha            3 pCi/l in water

Gross Beta            6 pCi/l in water

This precision at the 95% confidence level is approximately 24% for gross alpha and 18% for gross beta.

D. Interferences

High dissolved solids will contribute high statistical error.

Radiation lost by self-absorption;

Gross Alpha:            Greater than  $5.5 \text{ mg/cm}^2$ .

Gross Beta:            Greater than  $10 \text{ mg/cm}^2$ .

E. Analysis Rate

40 water samples per technician per 8 hour day.

II. Chemistry

- A. Gross alpha and gross beta measurements do not determine uniquely the presence of any particular nuclide. Instead, the gross activity of all alpha and beta emitters, respectively, are measured.

B. N/A

C. N/A

### III. Apparatus

- A. Instrumentation - Low background internal gas flow proportional counter having alpha background of 0.2 cpm or less and beta background of 2.5 cpm or less. A Beckman Wide Beta II Low Background Proportional System is used for this method.

The gas proportional detectors have thin windows which are connected to anticoincidence detectors. The anticoincidence detectors eliminate all radiation pulse from outside radiation.

B. Parameters

Alpha plateau

Beta plateau

C. Hardware/Glassware

1. 10 ml and 500 ml graduated cylinders
2. 400 ml beakers
3. Rubber policeman
4. Precleaned two (2) inch stainless steel planchets
5. Desiccator to hold planchets

D. Chemicals

1. Concentrated (16 N) nitric acid ( $\text{HNO}_3$ )
2. Dilute (0.5 N) nitric acid - 32 ml concentrated nitric acid diluted to 1.0 liter with deionized water
3. 30% hydrogen peroxide ( $\text{H}_2\text{O}_2$ )

### IV. Standards

A. Calibration Standards

Gross Alpha: Plutonium-239 NBS certified standard

Gross Beta: Strontium-90 and Cesium-137 NBS certified standards

B. Control Spikes

A known activity is added to one (1) liter of water and issued to the technician. The spike is introduced at a frequency of 10%.

## V. Procedure

### A. Sample Analysis

1. Transfer a 250 ml aliquot of the unfiltered samples to a 400 ml beaker.
2. Add 10 ml concentrated nitric acid and evaporate to dryness.
3. Ash with approximately 5 ml concentrated nitric acid and 5 ml 30% hydrogen peroxide.
4. Repeat ashing until residue is white to grayish white.
5. Dissolve the residue in a minimum volume of 0.5 N  $\text{HNO}_3$  and transfer to a tared 2" stainless steel planchet. Evaporate to dryness at low heat. Do not allow to splatter.
6. Rinse the beaker three (3) times with 5 ml of 0.5 N  $\text{HNO}_3$ ; poling the beaker walls and bottom, and transfer each wash to the planchet.
7. Evaporate to dryness, cool and reweigh.
8. Calculate the total residue present in order to correct for self absorption.
9. Count planchet utilizing instrumentation stated in Section III, A.

### B. Instrument Calibration Performed

Using NBS certified standards (see Section IV, A).

## VI. Calculations

### Alpha and Beta Calculations

Fill in data on Rad Chem Sheet

Time

Aliquot (in grams or liters)

Sample counts

Background counts (from reagent blank, if no blank, then use a SS blank)

Alpha efficiency = Find weight of sample in mg, and divide that by  $19.62 \text{ cm}^2$  (area of planchette) - the number you get is in  $\text{mg/cm}^2$

Go to graph and find corresponding efficiency.

Minimum eff = .02    Maximum = .23



Beta efficiency = Use the latest Beta Sr,Y-90 efficiency for the instrument sample was counted on.

Decay = 1

Recovery = 1

$$\text{Activity} = \frac{(\text{sample counts}) - (\text{background counts})}{(\text{time})(\text{eff.})(\text{aliquot})(\text{decay})(\text{rec.})(2.22)} = \text{pCi/aliquot}$$

$$\text{Statistics} = \frac{1.96 \sqrt{\frac{\text{sample counts} + \text{background counts}}{\text{Time}^2}}}{(\text{eff.})(\text{aliquot})(\text{decay})(\text{recovery})(2.22)} = \text{pCi/aliquot}$$

#### VII. Reference

Southwestern Radiological Health Laboratory Handbook of Radiochemical Analytical Methods, Third Reprint, March 1973.  
14th Edition, Wastewater Manual, Part 300.

## F-11.0 DETERMINATION OF NITROGLYCERINE AND PETN IN WATER BY HPLC (QUALITATIVELY)

I. Application: This method is designed to qualitatively determine the concentration of nitroglycerine and PETN in water.

A. Tested Concentration Range: ( $\mu\text{g/L}$  in water)

Nitroglycerine	- 5 to 50 $\mu\text{g/L}$
PETN	- 5 to 50 $\mu\text{g/L}$

B. Sensitivity: Response (integrator peak height) at the detection limit

Nitroglycerine	- 2000
PETN	- 900

C. Detection Limit: ( $\mu\text{g/L}$  in water)

Nitroglycerine	- 12
PETN	- 5

D. Interferences: None observed

E. Analysis Rate: A set of six samples can be extracted, dried and diluted in about three hours. LC analysis required 12 minutes per sample.

## II. Chemistry

A. Alternate Nomenclature and Chemical Abstracts Registry Number

Nitroglycerine: 1,2,3-propanetriol trinitrate; CAS RN 55-63-0

PETN: Pentaerythritoltetranitrate; 2,2-bis[(nitrooxy)-methyl]-1,3-propanediol dinitrate (ester); CAS RN 78-11-5

B. Physical and Chemical Properties

Nitroglycerine:  $\text{C}_3\text{H}_5\text{N}_3\text{O}_9$ ; M.W. 227.09 g/mol; Explodes at  $218^\circ\text{C}$

PETN:  $\text{C}_5\text{H}_8\text{N}_4\text{O}_{12}$ ; M.W. 316.15 g/mol; m.p. =  $140^\circ\text{C}$

These compounds are explosive. Use caution in handling. Potential toxic inhalation and skin absorption hazards exist.

C. Chemical Reactions: N/A

### III. Apparatus

A. Instrumentation: Spectra-Physics SP-8700 ternary solvent delivery system, Spectra-Physics SP-4100 computing integrator, Perkin-Elmer Model LC-75 UV-Vis variable wavelength detector with Model LC-75 autocontrol, Waters WISP Model 710 B autosampler.

B. Parameters

1. Column - Perkin Elmer Silica A/10, 0.26 cm x 25 cm
2. Mobile Phase - 0.3% isopropanol/99.7% isooctane
3. Flow Rate - 2 ml per minute
4. Detector - 204 nm
5. Injector Volume - 175  $\mu$ l

C. Hardware/Glassware

1. Volumetric Flasks: 10 ml, 50 ml, 100, ml, 1000 ml
2. Screw-cap tubes, 20 ml with Teflon-lined screwcaps
3. Graduated Cylinder, 100 ml
4. Separatory Funnels, 250 ml
5. Pipets: 1 ml and 5 ml
6. Pasteur pipets
7. Evaporator
8. Autosampler vials with Teflon-faced septa
9. Syringe: 250  $\mu$ l

D. Chemicals

1. Nitroglycerine and PETN SARM's
2. Isopropanol, HPLC grade
3. Hexane, HPLC grade
4. Water, HPLC grade
5. Methylene Chloride
6. Acetonitrile, HPLC grade
7. Nitrogen gas for drying
8. 2-nitrodiphenylamine, reagent grade

E. Reagents: N/A

### IV. Standards

A. Calibration Standards

1. Concentrated stock solutions are made by measuring 25 mg of each SARM into separate 25 ml volumetric flasks and diluting to final volume with isopropanol which is 0.05 mM in 2 nitrodiphenylamine. This results in solutions of nitroglycerine and PETN each 1 mg/ml. These solutions are stored protected from light under refrigeration.

2. A dilute stock solution is prepared daily by adding 125  $\mu$ l of each of the concentrated stocks to the same 50 ml volumetric flask and diluting to volume with 0.3% isopropanol/99.7% isooctane. This results in a solution that is 2.5  $\mu$ g/ml in each analyte. It is used as a high standard.
3. Working standards are prepared by adding the appropriate volumes of the dilute stock solution to 10 ml volumetric flasks and diluting to volume with 0.3% isopropanol/99.7% isooctane as indicated below:

<u>Volume (ml) of Dilute Stock Solution (2.5 <math>\mu</math>g/ml)</u>	<u>Final Volume</u>	<u>Final Concentration of Each Analyte</u>
0	10 ml	0
0.5	10 ml	0.125 $\mu$ g/ml
1.0	10 ml	0.250 $\mu$ g/ml
2.0	10 ml	0.500 $\mu$ g/ml
5.0	10 ml	1.25 $\mu$ g/ml
10.0	10 ml	2.50 $\mu$ g/ml

#### B. Control Spikes

1. The concentrated stocks are used to prepare a stock spiking solution. One ml of each concentrated stock solution is added to the same 1000 ml volumetric flask and diluted to volume with water which is 0.05 mM in 2-nitrodiphenylamine. The resulting solution is 1  $\mu$ g/ml in each of the analytes.
2. The control spikes are prepared by adding the appropriate volumes of stock spiking solution to 100 ml volumetric flasks and diluting to volume with water which is 0.05 mM in 2-nitrodiphenylamine as indicated below:

<u>Volume (ml) of Dilute Stock Solution (1 <math>\mu</math>g/ml)</u>	<u>Final Volume</u>	<u>Final Concentration of Each Analyte</u>
0	100 ml	0
250 $\mu$ l	100 ml	2.5 $\mu$ g/l
500 $\mu$ l	100 ml	5 $\mu$ g/l
1000 $\mu$ l	100 ml	10 $\mu$ g/l
2.5 ml	100 ml	25 $\mu$ g/l
5.0 ml	100 ml	50 $\mu$ g/l

- V. Procedure: Pour 100 ml of sample to be tested into a 250 ml separatory funnel and add 5 ml of methylene chloride which is 0.05 mM in 2-nitrodiphenylamine. Shake vigorously at least three minutes and allow to separate. Draw out extract with a Pasteur pipet and place into a 20 ml screw capped tube. Repeat procedure with another 5 ml of methylene chloride solution and combine the

extracts. Dry the extract under a stream of nitrogen. Add 2 ml of 0.3% isopropanol/99.7% isooctane and shake to insure that all of the sample is dissolved. Transfer contents to an autosampler vial. Sample is ready for LC analysis. The samples and standards are injected in aliquots of 175  $\mu$ l by autosampler.

- VI. Calculations: The integrator peak heights of the standards are plotted against the concentrations of the standards to obtain a standard curve. The apparent concentrations of the samples (in  $\mu$ g/ml) are obtained from the standard curve. The concentration of each analyte in the original sample is calculated as follows:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(\text{Apparent concentration in } \mu\text{g/ml}) \times 1000}{50}$$

VII. References

USATHAMA Method 6 B

## F-12.0 DETERMINATION OF TOTAL OIL AND GREASE IN WATER (QUALITATIVELY)

- I. Application: This method is designed to measure the fluorocarbon-113 extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the determination of relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter. Petroleum fuels from gasoline through #2 fuel oils are completely or partially lost in the solvent removal operation.
- A. Tested Concentration Range: 5 to 25 mg of extractable material per liter of water.
- B. Sensitivity: Milligrams
- C. Detection Limit: Milligrams calculated according to Hubaux and Vos.
- D. Interferences: Any extractable non-oily matter is an interference.
- E. Analysis Rate
- II. Chemistry:
- A. Alternate Nomenclature: The definition of oil and grease is based upon the procedure used, i.e., an oily material extractable in fluorocarbon 113.
- B. Physical and Chemical Properties: N/A
- C. Chemical Reactions: None
- III. Apparatus:
- A. Instrumentation: An analytical balance capable of weighing to 0.01 mg is used.
- B. Parameters: N/A
- C. Hardware/Glassware:
1. Separatory funnel, 2000 ml, with Teflon stopcock.
  2. Vacuum pump, or other source of vacuum.
  3. Flask, boiling, 125 ml (Corning No. 4100 or equivalent).
  4. Distilling head, Claisen or equivalent.
  5. Filter paper, Whatman No. 40, 11 cm.
  6. 100°C Oven
  7. Dessicator

D. Chemicals:

1. Hydrochloric acid, 1:1. Mix equal volumes of conc. HCl and distilled water.
2. Fluorocarbon-113, (1,1,2-trichloro-1,2,2-trifluoroethane), b.p.48°C.
3. Sodium sulfate, anhydrous crystal

IV. Standards:

- A. Calibration Standards: The balance calibration is maintained as part of the laboratory quality control program.
- B. Control Spikes: Wesson Oil is used as a spiking standard. The density of the Wesson Oil is determined by weighing an empty and filled 5 ml volumetric flask. The following control spikes are prepared by adding the indicated amounts of Wesson Oil to one liter samples of water with a syringe:

<u>mg Wesson Oil</u>	<u>Concentration mg/L</u>
0	Blank
5	5
10	10
25	25

- V. Procedure: Mark the bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 5 ml of 1:1 hydrochloric acid to the sample bottle. After mixing the sample, check the pH by touching pH-sensitive paper to the cap to insure that the pH is 2 or lower. Add more acid if necessary.

Pour the sample into a separatory funnel.

Tare a boiling flask (pre-dried in an oven at 103°C and stored in a desiccator).

Add 30 ml fluorocarbon-113 to the sample bottle to rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate, and filter the solvent layer into the flask through a funnel containing solvent moistened filter paper.

NOTE: An emulsion that fails to dissipate can be broken by pouring about 1 g sodium sulfate into the filter paper cone and slowly draining the emulsion through the salt. Additional 1 g portions can be added to the cone as required.

Repeat the rinse and extraction twice more, with additional portions of fresh solvent combining all solvent in the boiling flask.

Rinse the tip of the separatory funnel, the filter paper, and then the funnel with a total of 10-20 ml solvent and collect the rinsings in the flask.

Connect the boiling flask to the distilling head and evaporate the solvent by immersing the lower half of the flask in water at 79°C. The solvent may be collected for re-use. A solvent blank should accompany each set of samples.

When the temperature in the distilling head reaches 50°C or the flask appears dry remove the distilling head. Sweep out the flask for 15 seconds with air to remove solvent vapor by inserting a glass tube connected to a vacuum source. Immediately remove the flask from the heat source and wipe the outside to remove excess moisture and fingerprints. Cool the boiling flask in a desiccator for 30 minutes and weigh.

V. Calculations:

$$\text{mg/l total oil and grease} = \frac{R - B}{V}$$

where:

R = residue, gross weight of extraction flask minus the tare weight, in milligrams.

B = blank determination, residue of equivalent volume of extraction solvent, in milligrams.

V = volume of sample, determined by refilling sample bottle to calibration line and correcting for acid addition if necessary, in liters.

VII. References:

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979, Method 413.1.



APPENDIX G  
Quality Control Reports

July 29, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: AAA

Matrix: Leach


Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

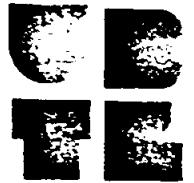
Cl	1.06
F	1.08
NO <sub>3</sub>	0.901
NO <sub>2</sub>	0.838
PO <sub>4</sub>	0.989
SO <sub>4</sub>	1.15

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: Chloride was detected in the field blank above the level of the quality control sample. However, the level was insignificant when compared with the levels detected in all other field samples. No other significant contamination was detected.

Evaluation: The results of the analysis of the quality control sample and of the field samples are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

A DIVISION OF  
THE UNIVERSITY OF UTAH  
RESEARCH INSTITUTE  
  
MEDICINE  
BIOENGINEERING  
CHEMISTRY  
  
RESEARCH  
DEVELOPMENT  
ANALYSIS

July 29, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: AAB

Matrix: Leach

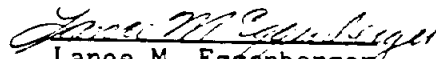
Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

Cl	1.16
F	1.23
NO <sub>3</sub>	0.937
NO <sub>2</sub>	0.877
PO <sub>4</sub>	1.04
SO <sub>4</sub>	1.23

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: A slight contamination of chloride was detected in the field blank. No other contaminants were detected.

Evaluation: The chloride contamination in the blank was not significant when compared with the chloride levels detected in the other field samples. The analysis of the quality control sample is acceptable, as is the analysis of the field samples.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
520 WAKARA WAY  
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801 581-8267

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August 6, 1982



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SALT LAKE CITY,  
UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Nitroglycerin and PETN

Lot: AAC

Matrix: Leach

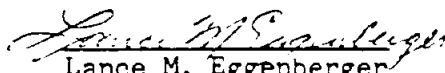
Standard Q.C.: The analysis of the quality control samples resulted in the following recoveries:

Nitroglycerin	1.12
PETN	0.704

Natural Water Spike: No natural water spike sample was included in this lot of leach samples.

Blanks: No significant contamination was detected in the field blank samples.

Evaluation: The analysis of the quality control sample is acceptable. No nitroglycerin was detected in the field samples. No PETN was detected in the field samples above the quantity detected in the quality control sample. All field sample PETN values were therefore below the limit of detection. The analysis of the field samples is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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August 6, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Explosives

Lot: AAD

Matrix: Leach

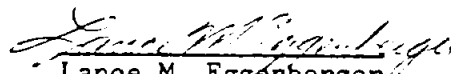
Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

24DNT	0.428
26DNT	0.33
246TNT	0.542
TETRYL	0.648
RDX	1.16

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: No contamination was detected in the blank field sample.

Evaluation: Although the recovery values of 24DNT, 26DNT, and 246TNT are below the anticipated values, when compared with other lots, the values of TETRYL and RDX are comparable. No compounds were detected in any of the field samples and therefore, even though the quality control sample recovery was low, no field sample contained compounds in concentrations higher than the limits of detection, as measured by the quality control sample. Therefore the analysis of the quality control sample is acceptable for screening purposes. The analysis of the field samples is also acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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June 4, 1982



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-6267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Semi-Volatiles

Lot: AAE

Matrix: Leach

Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

CL6ET	1.34
35DNA	0.767
DLDRN	0.883
24DNP	0.789

Natural Water Spike: No natural water spike was prepared for this lot.

Blanks: Two extraction method blanks were included in this lot. Two compounds were detected in the blanks. Only one field sample showed one compound at a value higher than in the blanks.

Evaluation: The recoveries of the compounds spiked in the quality control sample are acceptable. The blanks are also acceptable. Only one compound of interest was detected in one field sample.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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July 27, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Mercury

Lot: AAF

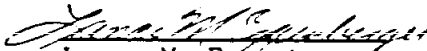
Matrix: Leach

Standard Q.C.: The analysis of the quality control sample resulted in a recovery of 0.932.

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blank: No significant contamination was detected in either of the field blanks.

Evaluation: The analysis of the quality control sample and of the field samples is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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July 28, 1982



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SALT LAKE CITY,  
UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals by ICP

Lot: AAG

Matrix: Leach

Standard Q.C.: The analysis of the quality control sample resulted in the following recoveries:

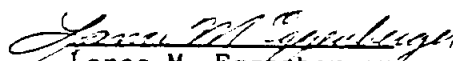
As	0.838
Be	1.87
Cd	0.962
Cr	0.827
Cu	0.972
Pb	1.04
Ni	0.859
Ag	0.51
Zn	0.00

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: A zinc contamination was detected in one of the two field blank samples.

Evaluation: The beryllium results in the quality control sample are slightly high. However, no beryllium was detected in any field samples. Therefore, the analysis of the quality control sample is acceptable for screening purposes.

The high blank result for zinc is suggestive of contamination. The sample was reanalyzed by graphite furnace with similar results, supporting the possibility of contamination. However, no zinc was detected in the quality control sample, for which no explanation is offered. The analytical results for zinc are open to question.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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July 29, 1982



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: AAH

Matrix: Leach

Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

Cl	1.12
F	0.944
NO <sub>3</sub>	1.06
NO <sub>2</sub>	0.949
PO <sub>4</sub>	0.924
SO <sub>4</sub>	1.14

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: No significant contamination was detected in the field blank sample.

Evaluation: The results of the analysis of the quality control sample and of the field sample are acceptable.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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July 27, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals - GF/AA

Lot: AAI

Matrix: Leach

Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

As	0.853
Ni	2.77
Zn	2.35

Natural Water Spike: No natural water spike sample was included with this lot.

Blanks: One field blank was high for nickel and both field blanks were high for zinc. Blanks showed no arsenic contamination.

Evaluation: The analysis results for arsenic are acceptable. The high analysis results for nickel and zinc in the quality control sample and the blanks are suggestive of contamination. A re-analysis of lot AAJ (the lot taken for sodium analysis from the same group of leach samples) for arsenic, nickel and zinc by GF/AA indicated that the contamination was likely to have been introduced before the instrumental analysis. Further investigation indicated that some of the nickel and zinc contamination come from the plastic sample containers, and some from the filtration apparatus required in the solid waste leaching procedure.

Subsequent to consultation with Dr. Les Eng (4/26/82), the following procedures were implemented:

1. All plastic sample bottles are rinsed with warm 50% redistilled nitric acid, rather than with cold 5% redistilled nitric acid.
2. The one liter plastic sample bottles, used for collection of samples to be analyzed for metals, are sent to the field without the nitric acid preservative in them. The preservative is added at the time of sample collection.



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August 6, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Sodium

Lot: AAJ


Matrix: Leach

Standard Q.C.: The analysis of the quality control sample resulted in a recovery of 1.22.

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: Both field blanks were evaluated as being slightly high in sodium content, averaging twice the value of the quality control sample.

Evaluation: The high blank values, plus the slightly high value for the analysis of the quality control sample, are suggestive of a slight sodium contamination. Two samples (AAJ004 and AAJ009) had analysis values at two to three times the average blank value. All other samples were sufficiently higher in sodium content as to be insignificantly affected by the blank values. The two low valued samples were sufficiently above the blank values to be evaluated, for screening purposes, as containing sodium at a concentration above the limit of detection for the analysis method. The results of the analysis of the quality control sample are acceptable for their purpose of screening samples and detecting possible contamination.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
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July 28, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Mercury

Lot: AAK


Matrix: Leach

Standard Q.C.: Analysis of the quality control sample resulted in a recovery value of 1.94.

Natural Water Spike: No natural water spike sample was prepared for this lot of leach samples.

Blank: No contamination was detected in the field blank sample.

Evaluation: The high value for the analysis of the quality control sample was the result of an instrumental problem. The problem was identified and repaired. The results of the analysis of the field samples are acceptable since they are all either well below the limit of detection, or zero. The analysis of the quality control sample is acceptable for screening purposes.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
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July 28, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: AAL

Matrix: Leach

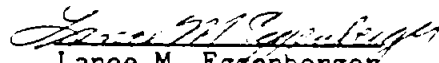
Standard Q.C.: The analysis of the quality control sample resulted in the following recoveries:

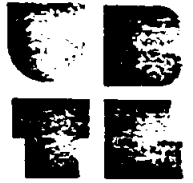
Cl	1.14
F	1.08
NO <sub>3</sub>	1.01
NO <sub>2</sub>	0.929
PO <sub>4</sub>	1.09
SO <sub>4</sub>	1.09

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: A slight contamination of chloride and of nitrate was detected in the field blank.

Evaluation: Analysis of the quality control sample is acceptable for all analytes. Although the blank showed slight contamination, it was much lower than was detected in the sample. The overall sample analysis is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Nitroglycerin and PETN

Lot: AAM

Matrix: Leach

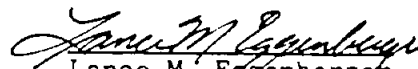
Standard Q.C.: Analysis of the Quality Control sample resulted in the following recoveries:

NG	0.542
PETN	0.889

Natural Water Spike: No natural water spike was included in this lot.

Blanks: No interferences or contamination were detected in the field blank

Evaluation: PETN showed very good recovery in the quality control sample. No PETN was found in any field sample. Although nitroglycerin (NG) recovery was 54% in the quality control sample, no NG was found in any field sample. Therefore, the analysis of the quality control sample for NG is acceptable as an indicator of proper analytical procedure. The overall analysis of both the field samples and the quality control sample is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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April 21, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: NG and PETN

Lot: AAN and ABB

Matrix: Soil Leach (AAN) and Water (ABB)

<u>Lot</u>	<u>NG</u>	<u>PETN</u>
AAN	1.04	.563
ABB	1.27	.765
Spike	.760	.665

The above values represent the corrected percent recovery for quality control samples in the indicated lots. Considering the vagaries of the analytical method, I consider these results acceptable. Although considered acceptable, it should be noted that PETN tends towards a lower than expected average recovery.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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August 6, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Explosives

Lot: AAO

Matrix: Leach


Standard Q.C.: Analyses of the quality control sample resulted in the following recoveries:

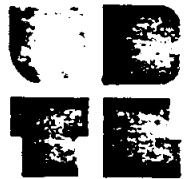
24DNT	0.782
26DNT	0.623
246TNT	0.759
TETRYL	0.241
RDX	0.535

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: No interferences or contamination was detected in the blank field sample.

Evaluation: Although the recovery of tetryl in the analysis of the quality control sample is lower than was anticipated, it is acceptable for screening purposes since no tetryl was detected in any of the field samples. The recoveries of the other analytes in the quality control sample are comparable to those of previous and of later analyses and are acceptable for screening purposes.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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August 6, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Explosives

Lot: AAP

Matrix: Leach

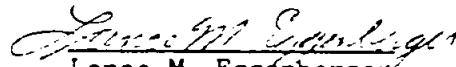
Standard Q.C.: analyses of the quality control sample resulted in the following recoveries:

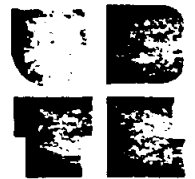
24DNT	0.452
26DNT	0.264
246TNT	0.823
TETRYL	0.614
RDX	0.482

Natural Water Spike: No natural water spike sample was included with this lot.

Blanks: No interferences or contamination was detected in the blank field sample.

Evaluation: Although the recovery of 26DNT in the analysis of the quality control sample is lower than was expected, it is acceptable for screening purposes since 26DNT was tentatively detected in only one field sample, which identification was later judged an interference. The recoveries of the other analytes in the analysis of the quality control sample are comparable to those of previous and of later analyses and are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
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June 4, 1982



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Semi-Volatiles

Lot: AAQ

Matrix: Leach


Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

CL6ET	1.83
35DNA	0.850
DLDRN	1.24
24DNP	0.782

Natural Water Spike: No natural water spike was prepared for this lot.

Blanks: Two extraction method blanks were included in this lot. Trace amounts of two compounds were detected in the blanks and in most of the samples.

Evaluation: The recoveries of the compounds spiked in the quality control sample are acceptable. Sample analysis, including blank correction, is acceptable. The blanks served to detect and quantitate some slight contamination, which did not appear in the quality control sample. The amount of contamination was not significant.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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August 6, 1982



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UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Sodium

Lot: AAR

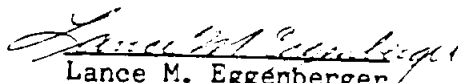
Matrix: Leach

Standard Q.C.: The analysis of the quality control sample resulted in a recovery of 1.016.

Natural Water Spike: No natural water spike sample was included with this lot of leach samples.

Blanks: One of the two field blank samples showed a level of sodium at twice that of the quality control sample. The other blank was evaluated at below the level of the quality control sample.

Evaluation: The results of the analysis of one field sample (AAR005) were comparable to the value of the high blank (AAR009). However, the lower blank (AAR004) was more closely related to the preparation and analysis of the sample than was the higher blank. The other field samples are sufficiently higher in value than the field blanks as to not constitute a significant problem or error. Therefore, the results of the analysis of the field samples are considered acceptable, as is the analysis of the quality control sample.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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July 27, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals - GF/AA

Lot: AAS

Matrix: Leach

Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

As	0.873
Ni	2.59
Zn	2.71

Natural Water Spike: No natural water spike sample was included with this lot.

Blanks: Both field blanks were high for nickel and for zinc. Blanks showed no arsenic contamination.

Evaluation: The analysis results for arsenic are acceptable. The high analysis results for nickel and zinc in the quality control sample and the blanks are suggestive of contamination. A re-analysis of lot AAJ (the lot taken for sodium analysis from the same group of leach samples) for arsenic, nickel and zinc by GF/AA indicated that the contamination was likely to have been introduced before the instrumental analysis. Further investigation indicated that some of the nickel and zinc contamination come from the plastic sample containers, and some from the filtration apparatus required in the solid waste leaching procedure.

Subsequent to consultation with Dr. Les Eng (4/26/82), the following procedures were implemented:


1. All plastic sample bottles are rinsed with warm 50% redistilled nitric acid, rather than with cold 5% redistilled nitric acid.
2. The one liter plastic sample bottles, used for collection of samples to be analyzed for metals, are sent to the field without the nitric acid preservative in them. The preservative is added at the time of sample collection.



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Dr. Les Eng suggested that data from this lot (AAS) be submitted for use in the survey subject to approval by the project officer, Mr. Don Campbell. Therefore, the analysis results are provisionally acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

May 27, 1982



UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals by ICP

Lot: AAT

Matrix: Leach

Standard Q.C.: Two quality control samples were prepared. One sample for silver only, and one for all metals except silver. The results of the analysis are as follows:

As	0.937
Be	1.12
Cd	0.893
Cr	0.951
Cu	0.847
Pb	1.00
Ni	0.730
Zn	0.976
Ag	0.861

Natural Water Spike: No natural water spike was included in this lot.


Blanks: Two leaching method blanks were included in this lot. These blanks showed no significant contamination. One of two analysis method blanks showed some silver contamination during analysis. Two field sample analyses also showed some silver contamination during one analysis run and were reanalyzed. The contaminated method blanks data was not used in sample evaluation.

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Evaluation: All quality control sample analyses were acceptable. The possibility of silver contamination was eliminated by reanalysis of the samples involved.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

May 5, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: AAU

Matrix: Water

Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in the following recoveries:

Cl	1.07
F	1.29
NO <sub>3</sub>	1.14
NO <sub>2</sub>	0.872
PO <sub>4</sub>	1.09
SO <sub>4</sub>	1.11

Natural Water Spike: Samples AAU006 and AAU007 were duplicate samples. AAU 006 was spiked at the same levels as was the QC sample. The analysis resulted in the following recoveries, including the initial value, that value corrected for the background (determined from the unspiked duplicate) and corrected for a 10% dilution factor resulting from spiking of the original solution.

<u>Anion</u>	<u>Uncorrected</u>	<u>Background Corrected</u>	<u>Dilution Corrected</u>
Cl	61.0	-12.7	-5.30
F	1.40	1.14	1.17
NO <sub>3</sub>	10.2	-0.17	0.864
NO <sub>2</sub>	0.026	0.0	0.0
PO <sub>4</sub>	0.752	0.668	0.677
SO <sub>4</sub>	26.0	-2.02	0.778



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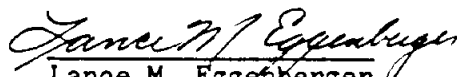
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Blanks: A laboratory blank analysis showed no significant anion contamination.

Evaluation: The standard QC sample analysis results are acceptable, although fluorine was slightly higher than previous results and nitrite was slightly lower than previous results.

In the natural sample spike, it appears that nitrite was completely eliminated in the solution, nitrate and phosphate were significantly reduced, fluoride was not affected, and chloride and sulfate were present in such high concentrations as to render the evaluation of the spiked amounts beyond the sensitivity of the analysis.



Lance M. Eggenberger  
Quality Assurance  
Specialist

July 28, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: AAV

Matrix: Water


Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

Cl	1.13
F	1.02
NO <sub>3</sub>	0.899
NO <sub>2</sub>	0.920
PO <sub>4</sub>	1.04
SO <sub>4</sub>	1.04

Natural Water Spike: No natural water spike sample was included with this lot.

Blanks: Nitrate was detected in the blank, near, but below the limit of detection value. No other analytes were detected in the blank in any significant quantity.

Evaluation: The analysis of the quality control sample is acceptable, as is the analysis of the field samples.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Volatiles

Lot: AAW

Matrix: Water

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Standard Q.C.: The analysis of the quality control sample resulted in the following recoveries:

CH3BR	1.43
CLC6H5	0.657
12DCLE	0.933

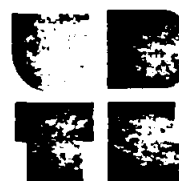
Natural Water Spike: No natural water spike was prepared for this lot.

Blanks: Blanks analyzed with this lot showed no significant interferences.

Evaluation: Recoveries from the quality control sample are within acceptable limits. All field sample values, for those compounds spiked in the quality control sample, were lower than in the quality control sample. Trace amounts of TRCLE were found in the quality control sample. Significantly larger amounts of TRCLE were recovered from three of four N-2 field samples. The three amounts were very comparable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Volatiles

Lot: AAX

Matrix: Water

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Standard Q.C.: The analysis of the quality control sample resulted in the following recoveries:

CH3BR	2.48
CLC6H5	0.978
12DCLE	1.10

Natural Water Spike: No natural water spike was prepared for this lot.

Blanks: All blank values were well below the L.O.D.

Evaluation: CH3BR recovery was 0.912 before slope correction. No CH3BR was detected in any field sample. The analysis of both the quality control and the field samples is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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April 23, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Mercury

Lot: AAY

Matrix: Water

Standard QC: The quality control sample was prepared as a water sample. The analysis resulted in a recovery of 4.11. Standards were evaluated and a correction factor (.61) was applied. The corrected recovery was 2.50.

Natural Water Spike: It is not recorded in the QC log whether a natural spike was made. However, the duplicate samples (AAY 002 and AAY 004) demonstrate very different amounts of mercury. AAY 002 is blank (0.0) while AAY 004 shows .220  $\mu\text{g/L}$  (uncorrected). If AAY 004 was spiked, its corrected recovery would be 1.19. With standards correction recovery was .73.

Blanks: The blank, AAY 007 showed a negative value.

Evaluation: It was established, after sample analysis, that the standards were low. This produced high QC results. A correction factor was insufficient to bring the QC value within acceptable limits. The evaluation of a natural spike is uncertain. However, all natural samples were below the QC sample. If the only major source of error is accepted as being in the standards, then the samples could be judged as being below the L.O.D. From the analytical results presented, it seems justified to accept this evaluation of the field samples.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist

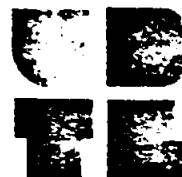


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# QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

## Reference Data:

Analysis of: Semi-Volatiles

Lot: AAZ

Matrix: Water

Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

CL6ET	1.76
35DNA	1.15
DLDRN	0.865
24DNP	0.591

Natural Water Spike: Samples AAZ006 and AAZ007 were duplicate samples. AAZ006 was spiked at the same levels as was the QC sample. The analysis resulted in the following recoveries, including the initial value and that value corrected for the background, as determined from the unspiked duplicate:

	<u>Uncorrected</u> AAZ006	<u>Background</u> <u>Corrected</u> AAZ006
CL6ET	1.64	1.64
35DNA	1.81	1.81
DLDRN	0.962	0.962
24DNP	1.22	1.22

Blanks: The field blank (AAZ005) showed only a trace amount of DEP - 0.1 g/L. No other contamination was detected in the field blank.


Evaluation: The recoveries of the compounds contained in the standard quality control sample are acceptable and correlate well with previous recovery data. The recoveries of the compounds, spiked into the natural

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sample are also acceptable, although they generally tend to be higher than the standard QC sample recoveries. Compounds spiked into the natural sample were not detected in the duplicate natural sample.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

May 4, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: ABA

Matrix: Water

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Standard Q.C.: The analysis of the quality control sample resulted in the following recoveries:

Cl	1.04
F	1.05
NO <sub>3</sub>	1.38
NO <sub>2</sub>	1.01
PO <sub>4</sub>	1.06
SO <sub>4</sub>	1.02

Natural Water Spike: No natural water spike was prepared.

Blanks: No field blank was submitted.

Evaluation: Nitrate was slightly high in the QC sample, which may be attributed to slight contamination from nitric acid during the container washing process. All other analytes are acceptable.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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April 21, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: NG and PETN

Lot: AAN and ABB

Matrix: Soil Leach (AAN) and Water (ABB)

<u>Lot</u>	<u>NG</u>	<u>PETN</u>
AAN	1.04	.563
ABB	1.27	.765
Spike	.760	.665

The above values represent the corrected percent recovery for quality control samples in the indicated lots. Considering the vagaries of the analytical method, I consider these results acceptable. Although considered acceptable, it should be noted that PETN tends towards a lower than expected average recovery.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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May 3, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Five Explosives

Lot: ABC

Matrix: Water

Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in the following recoveries:

24DNT	.821
26DNT	.757
246TNT	.961
TETRYL	.998
RDX	.660

Natural Water Spike: Samples ABC003 and ABC006 were duplicate samples. ABC003 was spiked with a mixture of the five explosives at the same concentration as the QC sample. The analysis resulted in the following recoveries, both uncorrected and corrected for background, as determined from the unspiked duplicate sample.

	<u>Uncorrected</u>	<u>Background Corrected</u>
24DNT	.801	.801
26DNT	.735	.735
246TNT	.897	.897
TETRYL	.922	.922
RDX	.582	.582

Blanks: Blank values were determined with a standard (laboratory) blank. All values were zero. There was no field blank included in this set.



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Evaluation: All analysis values comparing the natural spike with the QC sample, are acceptably comparable. 246TNT and TETRYL show the best recoveries, RDX shows the lowest recovery. This set of samples shows, overall, the highest and most acceptable recovery for each explosive of all of the sets previously analyzed.

*Lance M. Eggenberger*

Lance M. Eggenberger

Quality Assurance

Specialist

May 4, 1982



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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anion  
Lot: ABD  
Matrix: Soil Leach and Water

Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in the following recoveries:

Cl	1.22
F	1.06
NO <sub>3</sub>	0.986
NO <sub>2</sub>	0.939
PO <sub>4</sub>	0.999
SO <sub>4</sub>	1.09

Natural Water Spike: Samples ABD005 and ABD008 were duplicate samples. ABD008 was spiked at the same levels as was the QC sample. The analysis resulted in the following recoveries, including the initial value, that value corrected for the background (determined from the unspiked duplicate), and corrected for a 10% dilution factor from spiking the original solution.

<u>Anion</u>	<u>Uncorrected</u>	<u>Background Corrected</u>	<u>Dilution Corrected</u>
Cl	No Evaluation possible >20,000 µg/L		
F	4.62	0.996	1.36
NO <sub>3</sub>	3.68	0.986	1.26
NO <sub>2</sub>	0.027	0.00	0.00
PO <sub>4</sub>	3.22	-0.317	0.036
SO <sub>4</sub>	No Evaluation possible >20,000 µg/L		

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Blanks: All field blanks showed no significant anion contamination.

Evaluation: The standard QC sample analysis results are acceptable, although chlorine was slightly higher than previous results.

In the natural sample spike, it appears that nitrite was completely eliminated in the solution and phosphate was significantly reduced. Precise evaluation of phosphate is difficult since the spiked sample was lower than the unspiked sample. Nitrate and fluoride were acceptable. Chloride and sulfate were present in such high concentrations as to render evaluation impossible.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

April 23, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Mercury  
Lot: ABE  
Matrix: Soil Leach and Water

Standard Q.C.: The quality control sample was prepared as a leach sample. The analysis resulted in a recovery of .761. Although this is lower than the first QC sample analyzed (lot AAF) it is much more acceptable than the results of more recent analyses (lot AAK-1.9, lot AAY-2.5).

Natural Water Spike: Samples ABE 018 and ABE 019 were duplicate water samples. ABE 018 was spiked with mercury as a QC sample. The analysis resulted in a recovery of 1.05, which, when corrected for the quantity of mercury in ABE 019, was 0.948. This recovery is acceptable.

Blanks: Several blank samples were analyzed with this Lot. Leach blanks showed a significant amount of mercury present, averaging .12 to .16 µg/L. Water blanks showed only .03 to .06 µg/L of mercury. The leach blanks seem to indicate a moderate amount of contamination from the leaching process.

Evaluation: The natural water spike is acceptable. Since the leach QC sample was not processed through the leaching procedure, it was not blank corrected. Although lower than expected, it is still acceptable. The blanks demonstrate that there is some contamination occurring in the leaching process.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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May 12, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Volatiles

Lot: ABF


Matrix: Water

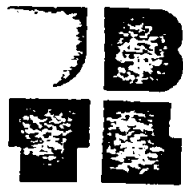
Standard QC: No standard QC sample was included in this lot.

Natural Water Spike: No natural water spike is performed on volatile samples.

Blanks No significant amounts (>L.O.D.) of reported compounds were detected in the blanks.

Evaluation: The omission of a standard QC sample from lot ABF was an oversight. Samples ABF 003 and ABF 004 were duplicate samples. T12DCE, found in ABF 003, was not found in ABF 004. Only 111-TCE was found at >1 µg/L, in sample ABF 004. All other results were <1 µg/L.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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# QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

## Reference Data:

Analysis of: Nitroglycerin and PETN

Lot: ABG

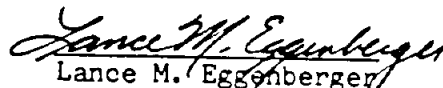
Matrix: Leach/Water

Standard Q.C.: The quality control sample was prepared as a water sample. The sample deteriorated prior to analysis. The decomposition products were clearly visible in the chromatogram.

Natural Water Spike: Samples ABG014 and ABG016 were duplicate natural water samples. ABG016 was spiked at the same levels as was the QC sample. The duplicate samples were of a contaminated surface water. Interferences present in the samples were of such character as to render analysis of the spiked sample impossible under the conditions of the analytical method (6B).

Blanks: All blank samples showed no contamination.

Evaluation: Although the standard quality control sample showed decomposition, no decomposition products, nor analytes of interest, were apparent in any of the samples. The natural water spikes were of no quality control value. The analysis of the field samples is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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May 20, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Five Explosives

Lot: ABH

Matrix: Leach/Water

Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in the following recoveries:

24DNT	0.900
26DNT	0.886
246TNT	0.849
TETRYL	0.475
RDX	0.671

Natural Water Spike: Samples ABH013 and ABH015 were duplicate samples. ABH013 was spiked with a mixture of the five explosives at the same concentration as the QC sample. The analysis resulted in the following recoveries, both uncorrected and corrected for background, as determined from duplicate sample ABH015.

	<u>Uncorrected</u>	<u>Corrected</u>
24DNT	0.0	0.0
26DNT	0.0	0.0
246TNT	0.0	0.0
TETRYL	2.83	0.792
RDX	3.53	1.77

Blanks: Field and extraction blanks showed no interferences.

Evaluation: The duplicate field sample had extensive interferences. These interferences rendered the sample unsuitable for a spike and the spike data uninformative.




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The standard QC sample, with no interferences is acceptable as an indicator of proper analysis.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

July 27, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Semi Volatiles

Lot: ABI

Matrix: Water/Leach

Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

CL6ET	0.869
35DNA	1.68
DLDRN	0.757
24DNP	1.21

Natural Water Spike: Samples ABI017 and ABI017 and ABI 018 were duplicate samples. ABI017 was spiked at the same levels as was the QC sample. The analysis resulted in the following recoveries, including the initial value and that value corrected for the background, as determined from the unspiked duplicate.

	<u>Uncorrected</u>	<u>Background Corrected</u>
	ABI017	ABI017
CL6ET	0.0	0.0
35DNA	0.0	0.0
DLDRN	0.0	0.0
24DNP	1.51	1.51

Blanks: No interferences or contamination were detected in any of the field blanks.

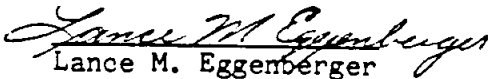
Evaluation: The compounds spiked in the quality control sample and in the natural water sample were detected only in these two samples. These four compounds were not detected in any other field samples. The



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recoveries from the quality control sample are acceptable and are indicative of an acceptable overall analysis. Three of the four compounds spiked in the natural water sample were not quantitated due to masking interferences in the sample. The analysis of the compound which was detected is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

May 11, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals by GF/AA  
Lot: ABJ  
Matrix: Soil Leach and Water

Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in the following recoveries:

As	.788
Ni	3.23
Zn	1.29

Natural Water Spike: Two natural samples were spiked. ABJ004 (surface) and ABJ014 (well) were spiked at the same levels as the standard QC sample. Samples ABJ009 (surface) and ABJ008 (well) were duplicates of the spiked samples. The analysis resulted in the following recoveries, including the initial value and that value corrected for the background, as determined from the unspiked duplicate:

	<u>Uncorrected</u>		<u>Background Corrected</u>	
	<u>ABJ004</u>	<u>ABJ014</u>	<u>ABJ004</u>	<u>ABJ014</u>
As	0.487	0.540	0.378	0.729
Ni	8.09	3.77	6.35	2.71
Zn	30.90	2.05	-19.9	0.280



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Blanks: Blank values were as follows:

<u>Blank</u>	<u>Type</u>	<u>Corrected <math>\mu\text{g/L}</math></u>		
		<u>As</u>	<u>Ni</u>	<u>Zn</u>
ABJ002	Field	-1.53	18.8	2.17
ABJ007	Field	-1.37	6.34	2.78
ABJ020	Extraction	-1.17	8.51	1.43
ABJ024	Extraction	-1.86	-2.39	1.42
ABJ026	Extraction	-1.33	-0.65	0.937
<u>Spiking Level</u>		7.06	5.00	0.997
<u>Recovery Corrected</u>		7.73	5.32	0.898

Evaluation: As compared to previous analyses of these compounds in QC samples, arsenic was comparable, nickel was higher, and zinc was lower. In the natural spike samples arsenic was recovered at one-half of the standard QC sample recovery in the surface sample, and at the same recovery as the standard QC sample in the well water sample. Nickel was recovered at values equivalent to the QC values, and at two to three times the QC sample values. Zinc, in the natural sample spike, can not be evaluated. It was generally far below expected values, after back-ground correction.

The poor results in the analysis of the natural sample spikes is most probably due to contamination of the sample bottles from the cleaning process. Slight contamination was noted after these samples were collected. The cleaning procedure has been altered to eliminate the contamination from future samples. The contamination, principally of nickel and zinc, is very noticable in the evaluation of the blank samples.

The natural sample spikes have served the purpose of contamination detection and evaluation. The standard QC sample, as an evaluation of the analytical process, is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

May 3, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Sodium

Lot: ABK

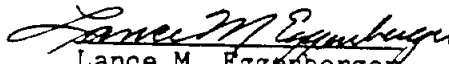
Matrix: Water

Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in a recovery of 0.921.

Natural Water Spike: Two natural samples were spiked. ABK003 (surface) and ABK015 (well) were spiked at a level of 997 µg/L. Samples ABK004 (surface) and ABK009 (well) were duplicates of the spiked samples. The analysis resulted in a recovery of 40.17, corrected for the duplicate value to 1.40, for sample ABK015 (well). Analysis of sample ABK003 (surface) resulted in a recovery of 503, corrected for the duplicate to -1.99.

Blanks: Blank values for the analysis were below the spiked QC values except for the special extraction blank #1000. It was 1.7 times the LOD value.

Evaluation: All QC spiked samples are within acceptable values. The natural spike samples, at 40 and 500 times the spike value, are considered of little significant value. It is very doubtful that the method is capable of distinguishing the amount of the spike in such a high background.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

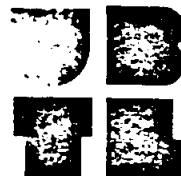


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# QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

## Reference Data:

Analysis of: Metals by ICP

Lot: ABL

Matrix: Leach/Water

Standard Q.C.: Two quality control samples were prepared. One sample for silver only, and one sample for all metals except silver. The analysis resulted in the following recoveries:

As	0.611
Be	1.87
Cd	1.03
Cr	0.827
Cu	1.57
Pb	0.860
Ni	0.752
Zn	0.903
Ag	0 (0.58 with background correction)

Natural Water Spike: Two natural samples were spiked. ABL013 (well) and ABL025 (surface) were spiked at the same levels as the QC sample in all metals except silver. No natural samples were spiked with silver. Samples ABL012 (well) and ABL024 (surface) were duplicates of the spiked samples. The analysis resulted in the following recoveries, including the initial value, and that value corrected for the background, as determined from the unspiked duplicate:

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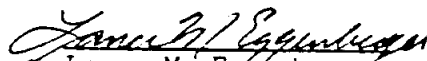


	<u>Uncorrected</u>		<u>Background Corrected</u>	
	ABL013	ABL025	ABL013	ABL025
As	0.747	1.34	0.849	0.832
Be	1.50	2.62	1.50	1.31
Cd	0.859	5.67	0.859	0.52
Cr	4.76	62.9	0.62	1.03
Cu	1.57	10.19	0.941	0.63
Pb	4.76	36.2	0.967	0.430
Ni	0.806	1.40	0.752	0.591
Zn	1.26	3.85	0.783	-5.06

Blanks: Neither field blanks nor leach method blanks showed any significant interferences or contamination.

Evaluation: The results of the analysis of the quality control sample are generally acceptable. Copper tended to be higher than in previous analyses, while arsenic and lead tended to be slightly lower than in previous analyses. Silver, when corrected for a negative background, (which was an artifact of the ICP), corresponded with previous analyses, as did all remaining elements in the QC sample.

The overall analysis of the spiked natural field samples is acceptable. When corrected for background, as determined from analysis of the duplicate sample, the analysis of the well water sample was overall better than the analysis of the surface water sample. This was probably due mainly to the much higher element concentrations present in the surface water sample.

  
 Lance M. Eggenberger  
 Quality Assurance  
 Specialist

May 12, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Cyanide

Lot: ABM

Matrix: Water

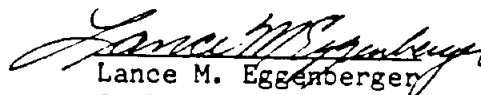
Standard Q.C.: The analysis of the QC sample resulted in a recovery of 0.719.

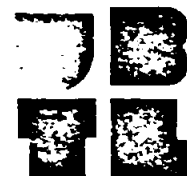
Natural Water Spike: Two natural samples were spiked. ABM008 (well) and ABM014 (surface) were spiked at a level of 10.36  $\mu\text{g/L CN}^-$ . Samples ABM007 (well) and ABM013 (surface) were duplicates of the spiked samples. The analysis resulted in the following recoveries, including the initial value and that value corrected for the background, as determined from the unspiked duplicate:

	<u>Uncorrected</u>		<u>Background Corrected</u>	
	ABM008	ABM014	ABM008	ABM014
CN <sup>-</sup>	0.842	4.400	0.880	1.32

Blanks: No significant amounts were detected in the blanks.

Evaluation: The QC sample and the natural water spikes are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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May 12, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Oil and Grease

Lot: ABN


Matrix: Water

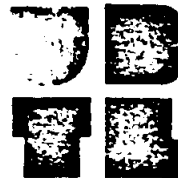
Standard Q.C.: No QC sample was included with this lot.

Natural Water Spike: No spike was performed in this lot.

Blanks: Blanks were below the L.O.D.

Evaluation: Duplicate samples with values above the L.O.D. are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



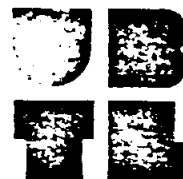
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May 12, 1982



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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anion

Lot: ABO

Matrix: Water

Standard Q.C.: The analysis of the quality control sample resulted in the following recoveries:

Cl	0.851
F	1.01
NO <sub>3</sub>	0.951
NO <sub>2</sub>	0.912
PO <sub>4</sub>	1.06
SO <sub>4</sub>	1.07

Natural Water Spike: No natural water spike was included in this lot.

Blanks: No field blank was included in this lot.

Evaluation: The recovery of chloride was lower than expected. All previous recoveries have been above 1.00. However, all field samples were >20,000 µg/L. All other analytes are acceptable.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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May 20, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: ABP

Matrix: Water

Standard Q.C.: Two QC samples were included with this lot. ABP004 (QC0043) was analyzed and the results were not acceptable. QC0055 was included with the set, and the entire set was reanalyzed. Recoveries are as follows:

	<u>QC0043</u>	<u>QC0055</u>	
	<u>First Analysis</u>	<u>Second Analysis</u>	<u>Only Analysis</u>
Cl	>20,000 µg/L	1.10	1.03
F	1.01	0.759	0.743
NO <sub>3</sub>	1.32	1.15	1.32
NO <sub>2</sub>	0.017	0.948	0.984
PO <sub>2</sub>	0.687	0.878	0.930
SO <sub>4</sub>	>20,000 µg/L	0.948	0.893

Natural Water Spike: No natural water spike was included with this set.

Blanks: No field blanks were analyzed with this lot.

Evaluation: Problems of contamination and analyte loss which were evidenced in the first analysis of QC0043 were not seen in the reanalysis of the sample. The results of the analysis of the additional




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QC sample (QC0055) agreed very closely with the first sample reanalysis results. The results, obtained from the second analysis of the lot, are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

May 13, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: ABQ

Matrix:

Standard Q.C.: The analysis of the QC sample resulted in the following recoveries:

Cl	2.79
F	0.817
NO <sub>3</sub>	1.33
NO <sub>2</sub>	0.818
PO <sub>4</sub>	1.09
SO <sub>4</sub>	2.27

Natural Water Spike: No natural water spike was included in this lot.

Blanks: No field blank was included in this lot.

Evaluation: A slight contamination of chloride and sulfate was present, making these anion's recovery high. However, all samples were far above measurable limits. All other anions were acceptable.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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May 20, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Cyanide

Lot: ABR


Matrix: Water

Standard Q.C.: The analysis of the QC sample resulted in a recovery of 0.654.

Natural Water Spike: No natural water spike was included in this lot.

Blanks: A reagent blank showed no interference.

Evaluation: The quality control sample was slightly (10%) lower than the QC sample analyzed with the next previous set (ABM). Although low, the quality control sample was much higher than the highest valued field sample (ABR002 at 0.98  $\mu\text{g/L}$ ) and is therefore considered acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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May 12, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Mercury

Lot: ABS

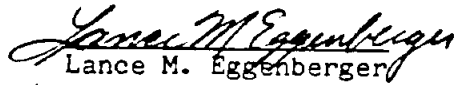
Matrix: Water

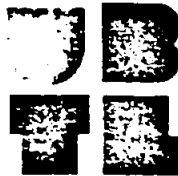
Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in a recovery of 0.645.

Natural Water Spike: No natural water spike was included in this lot.

Blanks: No field blanks were included with this analysis. Method blank values were below the LOD and below the QC value.

Evaluation: Although the QC sample recovery value was lower than for previous analysis, all field samples were lower than the QC sample. The QC sample analysis is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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July 20, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Semi Volatiles

Lot: ABT

Matrix: Water

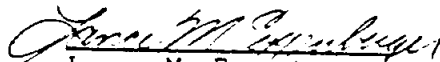
Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

CL6ET	0.609
35DNA	0.405
DLDRN	1.047
24DNP	0.476

Natural Water Spike: No natural water spike was included with this lot.

Blanks: No interferences or contamination were detected in any of the field blanks.

Evaluation: Although the recoveries of CL6ET, 35DNA, and 24DNP are lower than obtained in previous analyses, the results are acceptable, since none of these compounds were detected in any field sample. The recovery of DLDRN is acceptable. The analysis of the quality control sample is indicative of the acceptable functioning of the extraction and analysis procedure.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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May 21, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Volatiles

Lot: ABU

Matrix: Water

Standard Q.C.: The analysis of the quality control sample resulted in the following recoveries:

	<u>Slope Corrected</u>	<u>Without Slope Correction</u>
CH3BR	4.78	1.753
CLC6H5	0.731	1.016
12DCLE	1.25	0.956

Natural Water Spike: No natural water spike was prepared for this lot.

Blanks: Method blanks showed no significant interferences.

Evaluation: All spiked compounds were found in the quality control sample. No compounds were detected in the field samples at levels above the QC spiked levels. The QC analysis is acceptable. It should be noted that without slope correction, all QC values were nearer to the spiked value than with slope correction.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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# QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

## Reference Data:

Analysis of: Five Explosives

Lot: ABV

Matrix: Water

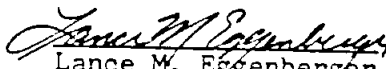
Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

24DNT	0.782
26DNT	0.779
246TNT	0.802
TETRYL	0.476
RDX	0.463

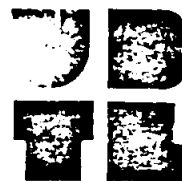
Natural Water Spike: No natural water spike was prepared with this lot.

Blanks: No interferences were detected in the laboratory method blank.

Evaluation: No analytes of interest were found in any of the field samples. Recoveries in the analysis of the quality control sample are consistant with previous quality control sample analyses. The analysis of the field samples and of the quality control sample are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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# QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

## Reference Data:

Analysis of: Nitroglycerin and PETN

Lot: ABW

Matrix: Water

Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in the following recoveries:

Nitroglycerin	0.352
PETN	0.926

Natural Water Spike: No natural water spike was included in this lot.

Blanks: No interferences were observed in the method blank.

Evaluation: Although nitroglycerin recovery was lower than in most previous quality control sample analyses, significant amounts of nitroglycerin were not detected in any sample. PETN recovery in the quality control sample analysis was better than in any previous QC sample. PETN was detected in only one field sample in any reportable amount. However, it was much lower than in the quality control sample. The analysis of the quality control sample is acceptable.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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May 20, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS


Reference Data:

Analysis of: Oil and Grease

Lot: ABX

Matrix: Water

No quality control is performed on oil and grease analysis.

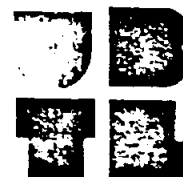
  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Sodium

Lot: ABY


Matrix: Leach

Standard Q.C.: The analysis of the quality control sample resulted in a recovery of 0.936.

Natural Water Spike: No natural spike was included with this lot.

Blanks: No sodium was detected in the blank sample.

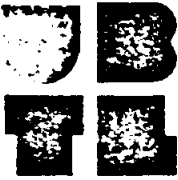
Evaluation: The result of the quality control sample analysis was within acceptable limits.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals - GF/AA

Lot: ABZ

Matrix: Water


Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

As	0.763
Ni	0.904
Zn	1.57

Natural Water Spike: No natural water spiked was prepared for this lot.

Blanks: No interferences or contamination were detected in the field blank.

Evaluation: The recoveries of the quality control sample are acceptable. The nickel recovery is better than in any previous quality control sample analysis. Arsenic recovery is lower than in previous quality control sample analyses, though not significantly.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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August 11, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals-ICP

Lot: ACA

Matrix: Water

Standard Q.C.: Analysis of the quality control samples resulted in the following recoveries:

	ACA018	ACA019	ACA017	ACA020
As	0.933	0.950	---	---
Be	1.68	1.50	---	---
Cd	1.03	0.859	---	---
Cr	1.24	0.827	---	---
Cu	0.784	0.941	---	---
Pb	1.72	0.788	---	---
Ni	0.859	0.752	---	---
Ag	---	---	0.538	2.56
Zn	1.02	0.843	---	---

Natural Water Spike: No natural water spike sample was included in this lot.

Blanks: No contamination or interferences were detected in the blank.

Evaluation: Although beryllium appears to be high in both ACA018 and ACA019, the results are consistent with previous results. The results for lead are wide ranging but are acceptable for screening purposes. All field samples were either well above or well below the QC sample values, except ACA004, which was slightly above the theoretical limit of detection. Sample ACA004 should be accepted as containing lead at a concentration level equal to the limit of detection.

The results for silver analysis were also wide ranging. However, all field samples were either well above or well below the QC sample




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values, except ACA004, which was slightly above the theoretical limit of detection. Sample ACA004 should be accepted as containing silver at a concentration level equal to the limit of detection.

All other analyses are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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801 551-8267

# QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

## Reference Data:

Analysis of: Anions

Lot: ACB

Matrix: Leach

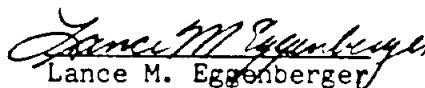
Standard Q.C.: The analysis of the QC sample resulted in the following recoveries:

Cl	1.03
F	1.07
NO <sub>3</sub>	1.00
NO <sub>2</sub>	0.862
PO <sub>4</sub>	1.00
SO <sub>4</sub>	1.04

Natural Water Spike: No natural water spike was included in this lot.

Blanks: No interferences or contamination were detected in the blank.

Evaluation: Although NO<sub>2</sub> is lower than the other analytes in the quality control sample, it is still well within acceptable limits for the analyte. No NO<sub>2</sub> was detected above the blank value in any field sample. The analyses of both the quality control sample and the field samples are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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ANALYSIS

August 2, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Semi Volatiles

Lot: ACC

Matrix: Leach

Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

CL6ET	0.327
35DNA	0.0
DLDRN	0.0
24DNP	0.0

Natural Water Spike: No natural water spike sample was included in this lot of leach samples.

Blanks: Only DEP was detected in the blank sample. It was at a lower level than was detected in any of the field samples.


Evaluation: The recovery of only one of four compounds from the quality control sample, plus the recovery of nine (9) additional compounds from the quality control sample, merited further investigation. The investigation showed that the mixture prepared for certification was spiked as the quality control sample instead of the four compound mixture normally used for quality control sample preparation. This accounts for the presence of nine (9) additional different compounds in the quality control sample.



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Although the wrong spike was used, the analysis of the quality control sample did show that the method was functioning. Therefore, the analysis of the quality control sample and of the field samples should be provisionally accepted.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

July 12, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Five Explosives

Lot: ACD

Matrix: Leach

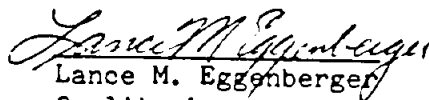
Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

24DNT	0.671
26DNT	0.650
246TNT	0.939
TETRYL	0.992
RDX	1.858

Natural Water Spike: No natural water spike was prepared with this lot.

Blanks: No interferences or contamination were detected in the field blank.

Evaluation: No analytes of interest were found in any of the field samples. Recoveries in the analysis of the quality control sample are generally consistent with previous quality control recoveries. RDX was much higher than expected, but no contamination was detected in either blanks or samples. However, the quality control sample and the field sample analyses are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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July 13, 1982



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UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Nitroglycerin and PETN

Lot: ACE

Matrix: Leach


Standard Q.C.: Analysis of the Quality Control sample resulted in the following recoveries:

NG	1.56
PETN	0.815

Natural Water Spike: No natural water spike was included in this lot.

Blanks: No interferences or contamination were detected in the field blank.

Evaluation: PETN showed acceptable recovery in the quality control sample. Nitroglycerin showed a recovery of 100.6% before application of the slope correction factor. After the correction factor was applied, the nitroglycerin recovery value (1.56) was slightly higher than previous recovery values. However, no nitroglycerin was detected in any sample.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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July 19, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Mercury

Lot: ACF

Matrix: Leach

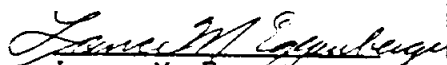
Standard Q.C.: The analysis of the quality control sample resulted in a recovery of 0.810.

Natural Water Spike: No natural water spike was included with this lot.

Blanks: No significant contamination was detected in the field blank.

Evaluation: The analysis for mercury in both quality control and field samples is acceptable.

NOTE: This data is the result of a re-extraction and re-analysis of the samples. The first analysis failed to pass the quality control requirements.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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July 12, 1982



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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Anions

Lot: ACG

Matrix: Water

Standard Q.C.: The analysis of the QC sample resulted in the following recoveries:

Cl	1.05
F	0.843
NO <sub>3</sub>	1.12
NO <sub>2</sub>	0.923
PO <sub>4</sub>	0.897
SO <sub>4</sub>	1.13

Natural Water Spike: Samples ACG002 and ACG005 were duplicate samples. ACG005 was spiked at the same levels as was the QC sample. The analysis resulted in the following recoveries, including the initial value, that value corrected for the background (determined from the unspiked duplicate), and corrected for a 10% dilution factor from spiking the original solution.

<u>Anion</u>	<u>Uncorrected</u>	<u>Background Corrected</u>	<u>Dilution Corrected</u>
Cl	No Evaluation Possible >20,000 µg/L		
F	2.34	0.730	0.891
NO <sub>3</sub>	3.39	0.620	0.896
NO <sub>2</sub>	0.18	0.00	0.016
PO <sub>4</sub>	0.213	0.00	0.021
SO <sub>4</sub>	No Evaluation Possible >20,000 µg/L		

Blanks: The field blanks showed no significant anion contamination.


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Evaluation: The standard QC sample analysis results are acceptable, although phosphate was slightly low than most previous results.

In the natural water sample spike it appears that nitrite and phosphate were essentially eliminated in the solution. Nitrate and fluoride were acceptable. Chloride and sulfate were present in such high concentrations as to render evaluation impossible.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

July 27, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Nitroglycerin and PETN

Lot: ACH

Matrix: Water

Standard Q.C.: The analysis of the quality control samples resulted in the following recoveries:

Nitroglycerin	1.32
PETN	0.689

Natural Water Spike: Samples ACH 001 and ACH 008 were duplicate water samples. ACH 001 was spiked at the same levels as the standard quality control sample. The analysis resulted in the following recoveries, including the initial value and that value corrected for the background, as determined from the unspiked duplicate sample.

	<u>Uncorrected</u>	<u>Background Corrected</u>
Nitroglucerin	1.04	1.04
PETN	0.00	0.00

Blanks: No contamination or interferences were detected in the field blank.

Evaluation: Results of the analysis of the quality control sample are comparable to previous results and are acceptable. Analysis of the natural spike sample is acceptable for nitroglycerin. PETN was not recovered in the natural water spike sample. Neither nitroglycerin nor PETN were detected in any field sample. The overall analysis is acceptable.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Five Explosives

Lot: ACI

Matrix: Water

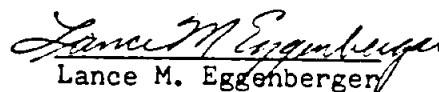
Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

24DNT	0.548
26DNT	0.481
246TNT	0.885
TETRYL	0.794
RDX	0.477

Natural Water Spike: No natural water spike was prepared with this lot.

Blanks: No contamination or interferences were detected in the field blank.

Evaluation: Although recovery for 24DNT and 26DNT were below the average previous recovery for each of these compounds these recoveries are acceptable because 24DNT and 26DNT were not observed in the field samples. The QC sample analysis results are overall acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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August 11, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals-ICP  
Lot: ACJ  
Matrix: Water/Leach

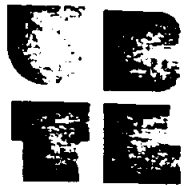
Standard Q.C.: The analysis of the quality control sample resulted in the following recoveries:

	<u>ACJ008</u>	<u>ACJ005</u>
As	1.00	---
Be	1.87	---
Cd	1.03	---
Cr	1.03	---
Cu	0.784	---
Pb	0.967	---
Ni	0.859	---
Ag	---	0.269
Zn	0.963	

Natural Water Spike: Samples ACJ006 and ACJ009 were duplicate samples. ACJ006 was spiked at the same levels as the QC sample in all metals except silver. No natural samples were spiked with silver. The analysis resulted in the following recoveries, including the initial value, and that value corrected for the background as determined from the unspiked duplicate:

	<u>Uncorrected</u>	<u>Background Corrected</u>
As	1.87	0.628
Be	2.06	0.749
Cd	0.859	0.515
Cr	2.07	0.207
Cu	2.67	1.57
Pb	0.752	0.430
Ni	0.752	0.430
Zn	2.83	1.32

Blanks: Only silver contamination was detected in the water blank. No other significant contamination was detected in either the water or the




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SALT LAKE CITY,  
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801 581-8267

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leach blanks.

Evaluation: The results of the analysis of the quality control sample are acceptable. Beryllium was high, comparable to its value in all previous analyses. Silver recovery was very low. However, except for the water blank, silver values in all field samples were below the QC sample value. Therefore, the QC sample results for silver are acceptable for screening purposes. The high silver results for the blank sample are unaccountable and therefore the field sample analysis for silver must be provisionally acceptable.

The overall analysis of the spiked natural field sample is acceptable. When corrected for background, as determined from the analysis of the duplicate sample, chromium was low and copper was slightly high, when compared with previous results. However, in both cases, the total amount of analyte in the duplicate samples was near the limit of detection and precise quantitation is difficult in natural matrix samples. therefore, for screening purposes, the natural spike sample is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

July 13, 1982



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Mercury

Lot: ACK

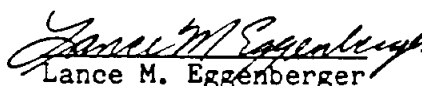
Matrix: Water/Leach

Standard Q.C.: Analysis of the Quality Control sample resulted in a recovery of 0.997.

Natural Water Spike: Samples ACK006 and ACK007 were duplicate samples. ACK007 was spiked at the same level as the QC sample. The result of the analysis of the spiked sample gave a recovery of 1.15 which, when corrected for background, as determined from the duplicate sample (ACK006), was 1.07.

Blanks: No significant interferences or contamination were detected in the blank.

Evaluation: Both the standard QC sample analysis results and the natural spiked sample analysis results are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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July 28, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Volatiles

Lot: ACL

Matrix: Water


Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

CH <sub>3</sub> BR	0.956
CLC <sub>6</sub> H <sub>5</sub>	1.04
12DCLE	1.05

Natural Water Spike: No natural water spike sample was included with this lot of volatile water samples.

Blanks: No contamination or interference was detected in the blank field sample.

Evaluation: The analysis of the quality control sample and of the field samples is acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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July 13, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Cyanide

Lot: ACM

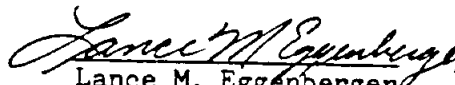
Matrix: Water

Standard Q.C.: Analysis of the Quality Control sample resulted in a recovery of 0.980.

Natural Water Spike: Samples ACM001 and ACM007 were duplicate samples. ACM007 was spiked at the same level as the QC sample. The uncorrected recovery was 1.65. When corrected for background, as determined from the unspiked duplicate sample, the recovery was 0.515.

Blanks: No significant amount of cyanide was detected in the field blank.

Evaluation: Recovery in the quality control sample was acceptable. The recovery in the spiked natural sample, while low after correction for background, was still acceptable. The spiked sample had the highest value of cyanide of all the samples analyzed.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist



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SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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August 2, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Semi Volatiles

Lot: ACN

Matrix: Water

Standard Q.C.: Analysis of the quality control sample resulted in the following recoveries:

CL6ET	0.858
35DNA	0.0
DLDRN	0.831
24DNP	0.0

Natural Water Spike: Samples ACN002 and ACN003 were duplicate samples. ACN002 was spiked with a mixture of the desired analytes at the same concentration as the quality control sample. The analysis resulted in the following recoveries, both uncorrected and corrected for background, as determined from duplicate sample ACN003.

	<u>Uncorrected</u>	<u>Background Corrected</u>
CL6ET	0.636	0.636
35DNA	0.0	0.0
DLDRN	1.01	1.01
24DNP	0.0	0.0

Evaluation: CL6ET and DLDRN were recovered from both the quality control sample and the natural water spiked sample. Subsequent analysis of the stock mixture used in spiking the quality control sample and the natural water spike sample showed that 24DNP had completely decomposed and 35DNA was below the limit of detection. These compounds were not recovered from the quality control sample nor the natural water spike.

The recovery of two of the spiked compounds and the accounting for



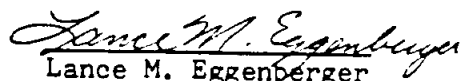
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the other two compounds lends credibility to the analysis of the lot. The detection of other non-quality control samples also lends credibility to the overall analysis. Based upon the overall results, the analysis is provisionally acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

July 13, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

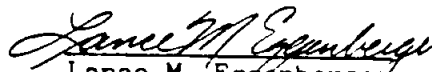
Reference Data:

Analysis of: Oil and Grease

Lot: ACO

Matrix: Water

Standard Q.C.: No quality control is performed on oil and grease analysis.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84103  
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July 19, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Metals - GF/AA

Lot: ACP

Matrix: Water/Leach

Standard Q.C.: The quality control sample was prepared as a water sample. The analysis resulted in the following recoveries:

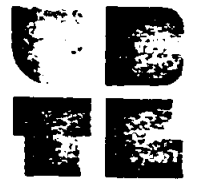
As	1.106
Ni	0.905
Zn	1.736

Natural Water Spike: Samples ACP012 and ACP013 were duplicate water samples. ACP012 was spiked at the same levels as the standard QC sample. The analysis resulted in the following recoveries, including the initial value and that value corrected for the background, as determined from the unspiked duplicate sample:

	<u>Uncorrected</u>	<u>Background Corrected</u>
	ACP012	ACP012
As	15.48	0.218
Ni	0.842	0.913
Zn	0.922	0.071

Blanks: There was no significant contamination for either arsenic or nickel in the field blanks. The blank values for zinc were comparable to the levels in the quality control sample and in the spiked natural sample and the duplicate of the spiked natural sample.

Evaluation: The analysis of the quality control sample is acceptable for all analytes. The analysis of the natural water spike is very good for nickel. Considering the high background found for arsenic, the




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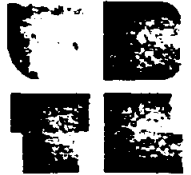
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determination of the spiked value is acceptable. The spiked value for zinc was essentially indistinguishable from the background. The uncorrected value was better than that value corrected for background. The spiked and blank values were below the values found in the other field samples. Therefore the analyses of the natural spike and the QC sample for zinc are acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

July 13, 1982



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520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:


Analysis of: Sodium  
Lot: ACQ  
Matrix: Water/Leach

Standard Q.C.: Analysis of the standard QC sample resulted in a recovery of 0.929.

Natural Water Spike: Samples ACQ010 and ACQ011 were duplicate samples. ACQ010 was spiked at the same level as was the QC sample. The analysis resulted in an initial recovery of 1387, or 1387 times the spiked amount. When this value was corrected for background, as determined from the duplicate sample, the recovery was 3.23.

Blanks: The field blanks showed no amounts of sodium present.

Evaluation: The standard QC sample analysis results are acceptable. Considering that the natural sample was spiked at a level of one part in 1400 (0.07%), a recovery of 3.23 times the spiked value (0.2%) is very acceptable.

  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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July 20, 1982

QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

Reference Data:

Analysis of: Gross alpha and beta

Lot: ACR

Matrix: Water/Leach

Standard Q.C.: Analysis of the quality control samples resulted in the following values:

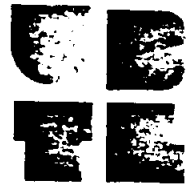
	ACR005	ACR056	ACR057
gross alpha	0.948	0.861	1.12
gross beta	1.23	1.18	1.36

Natural Water Spike: No natural water spike sample was prepared for this lot.

Blanks: No contamination was detected in the field blank samples.

Evaluation: The results of the analysis of the quality control samples are well within the precision and accuracy limits established by the analyst in reporting raw data with plus - minus values. The analysis of the quality control samples and of the field samples is acceptable.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist



UBTL  
520 WAKARA WAY  
SALT LAKE CITY,  
UTAH 84108  
801 581-8267

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SALT LAKE CITY,  
UTAH 84108  
801 581-8267

# QUALITY CONTROL REPORT

Submitted To: ERTEC, Tooele Survey

Submitted By: Lance M. Eggenberger, QAS

## Reference Data:

Analysis of: Gross alpha and beta

Lot: ACS

Matrix: Water/Leach

Standard Q.C.: Analysis of the quality control samples resulted in the following values:

	ACS016	ACS017
gross alpha	1.11	1.12
gross beta	1.34	1.36

Natural Water Spike: No natural water spike sample was prepared for this lot.

Blanks: No contamination was detected in the field blank samples.

Evaluation: The results of the analyses of the quality control samples are consistent with previous results and within the accuracy and precision limits as established by the analyst in reporting raw data with plus - minus values. The analysis of the quality control samples and of the field samples is acceptable.

*Lance M. Eggenberger*  
Lance M. Eggenberger  
Quality Assurance  
Specialist

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APPENDIX H  
Quality Control Results





JMR  
1.24

**Analyte:**

[illegible]



Rww  
6-5-9

Analyte:	35 DNA	Method:	3 W	Slope:	1.19
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PREPARED FOR ERTEC BY UBTL





Rww  
1.67

Analyte:	24 DNP	Method:	3W	Slope:	1.02
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PREPARED FOR ERTEC BY UBTL



Analyte:	<u>26DNT</u>	Method:	<u>2B</u>	Slope:	<u>0.741</u>
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PREPARED FOR ERTEC BY UBTL

Analyte: 246 TNT Method: 2B Slope: 0.852

PREPARED FOR ERTEC BY UBTL





Analyte: NG Method: 6B Slope: 0.643

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Analyte:	<u>PETN</u>	Method:	<u>6B</u>	Slope:	<u>1.08</u>
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Analyte:	AS	Method:	3T	Slope:	0.973
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PREPARED FOR ERTEC BY UBTL



Analyte:	CD	Method:	3T	Slope:	0.964
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Analyte:	<u>Cu</u>	Method:	<u>3T</u>	Slope:	<u>1.07</u>
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Analyte:	AG	Method:	3T	Slope:	0.929
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[illegible]



Analyte:	Z N	Method:	3T	Slope:	0.832
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Analyte: AS Method: 7T Slope: 0.913

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Analyte:	<u>NI</u>	Method:	<u>7T</u>	Slope:	<u>0.94</u>
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Analyte:	ZN	Method:	7T	Slope:	1.11
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PREPARED FOR ERTEC BY UBTL



Analyte:	NA	Method:	1M	Slope:	0.989
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UBTL QUALITY CONTROL SUMMARY FOR ERTEC/TOOELE SURVEY

Analyte: CL Method: 2P Slope: 0.971 LJD REB  
1.15

Chart #	Lab #	QC #	Type	Date Prepared	Date Analyzed	Target	Found	End./Tar.	F/T Slope	
1	AAH004	0001	LEACH	26 FEB	26 FEB	1000	1029	1.03	1.06	
2	AAB004	0002	LEACH	26 FEB	1 MAR	1000	1131	1.13	1.16	
3	AAH006	0009	LEACH	5 MAR	5 MAR	1000	1089	1.09	1.12	1.12
4	AAH001	0013	LEACH	11 MAR	11 MAR	1000	1106	1.106	1.14	1.12
5	AAH005	0023	Water	2 APR	2-3 APR	1000	1040	1.04	1.07	
6	AAV002	0024	Water	6 APR	6 APR	1000	1100	1.10	1.13	
7	ABA002	0029	Water	6 APR	7 APR	1000	1010	1.01	1.04	
8	ABD007	0032	Water	16 APR	16 APR	1000	1190	1.19	1.22	
9	ABD003	0042	Water	30 APR	30 APR	1000	926	.826	0.851	
10	ABP004	0043	Water	3 May	4 May	1000	1200	1.27	1.10	contaminated
11	ABR003	0044	Water	5 May	6 May	1000	3210	3.21	2.79	
	ABP---	0055	Water	12 May	13 May	1000	1190	1.19	1.03	(REB)
	ACB003	0056	Leach	4 June	5 June	1000	998	.998	1.03	(LSD)
	ACG006	0061	Water	25 JUN	25 JUN	1000	1210	1.210	1.052	(REB)

UBTL QUALITY CONTROL SUMMARY FOR ERTEC/TOOELE SURVEY

Analyte: F Method: 2P Slope: 0.894 LTD REB 1.15

Chart #	Lab #	QC #	Type	Date Prepared	Date Analyzed	Target	Found	End./Tar.	F/T Slope	
1	PAA004	0001	LEACH	26 FEB	26 FEB	1000	969	.969	1.08	
2	AAB004	0002	LEACH	26 FEB	1 MAR	1000	1101	1.10	1.23	
3	AAH006	0009	LEACH	5 MAR	5 MAR	1000	844 <sup>936</sup>	.844 <sup>936</sup>	1.08 <sup>1.05</sup>	REB 1.05 Action 4/2 1.32
4	AAL001	0013	LEACH	11 MAR	11 MAR	1000	963	.963	1.08	REB 1.05 Action 4/2 1.32
5	AAU005	0023	Water	2 APR	2-3 APR	1000	1150	1.150	1.29	
6	AAV002	0024	Water	6 APR	6 APR	1000	913	.913	1.02	
7	ABA002	0029	Water	6 APR	7 APR	1000	940	.940	1.05	
8	ABD007	0032	Water	16 APR	16 APR	1000	946	.946	1.06	
9	ABO003	0042	Water	30 Apr	30 Apr	1000	901	.901	1.01	
10	ABP004	0043	Water	3 MAY	4 MAY	1000	813 <sup>813</sup>	.813 <sup>813</sup>	1.01 <sup>1.01</sup>	REB 1.05
11	ABR003	0044	Water	5 MAR	6 MAY	1000	940	.940	.817	
	ABP---	0055	Water	12 May	13 May	1000	855	.855	.743	
	ACB003	0056	Leach	4 June	5 June	1000	956	.956	1.07	
	ACG006	0061	Water	25 JUN	25 JUN	1000	969	.969	.843 (REB)	



UBTL QUALITY CONTROL SUMMARY FOR ERTEC/TOOELE SURVEY

Analyte: N03 Method: 2P Slope: 1.14 LJD REB 1.06

Chart #	Lab #	QC #	Type	Date Prepared	Date Analyzed	Target	Found	End./Tar.	F/T Slope	
1	AA004	0001	LEACH	26 FEB	26 FEB	1005	1032	1.027	.901	
2	AB004	0002	LEACH	26 FEB	1 MAR	1005	1074	1.069	.937	
3	AP006	0009	LEACH	5 MAR	5 MAR	1005	1220	1.214	1.06	RETURN 1.41 2.10 1.60
4	AP001	0013	LEACH	11 MAR	11 MAR	1005	1160	1.15	1.01	RETURN 1.41 2.10 1.60
5	AA005	0023	Water	2 Apr	2-3 Apr	1005	1310	1.30	1.14	
6	AA002	0024	Water	6 Apr	6 Apr	1005	1030	1.025	.899	
7	ABA002	0029	Water	6 APR	7 APR	1005	1580	1.57	1.38	
8	ABD007	0032	Water	16 APR	16 APR	1005	1130	1.12	.986	
9	ABD003	0042	Water	30 Apr	30 Apr	1005	1090	1.08	.951	
10	ABP004	0043	Water	3 May	4 May	1005	1410	1.40	1.15	RETURN
11	ABQ003	0044	Water	5 May	6 May	1005	1420	1.413	1.33	
	ABP---	0055	Water	12 May	13 May	1005	1410	1.40	1.32	
	ACB003	0056	Leach	4 June	5 June	1005	1150	1.14	1.00	
	ACG006	0061	Water	25 JUN	25 JUN	1005	1190	1.184	1.117	REB

UBTL QUALITY CONTROL SUMMARY FOR ERTEC/TOOELE SURVEY

Analyte: N02 Method: 2P Slope: 1.05 LJD REB  
.956

Chart #	Lab #	QC #	Type	Date Prepared	Date Analyzed	Target	Found	Fnd./Tar.	F/T Slope
1	AAH004	0001	LEACH	26 FEB	26 FEB	995	875	.875	.838
2	AAB004	0002	LEACH	26 FEB	1 MAR	995	916	.921	.877
3	AAH006	0009	LEACH	5 MAR	5 MAR	995	991	.996	.949
4	AAH001	0013	LEACH	11 MAR	11 MAR	995	971	.976	.929
5	AAH005	0023	Water	2 APR	2-3 APR	995	911	.916	.872
6	AAV002	0024	Water	6 Apr	6 Apr	995	961	.966	.920
7	ABA002	0029	Water	6 APR	7 APR	995	1060	1.065	1.01
8	ABD007	0032	Water	16 APR	16 APR	995	981	.986	.939
9	ABD003	0042	Water	30 Apr	30 Apr	995	953	.958	.912
10	ABP004	0043	Water	3 May	4 May	995	901	.906	.848
11	ABD003	0044	Water	5 May	4 May	995	778	.782	.818
	ABP---	0055	Water	12 May	13 May	995	936	.941	.984
	ABP003	0056	Lead	4 June	5 June	995	901	.906	.862
	ACB006	0061	Water	25 JUN	25 JUN	995	878	.882	.923

UBTL QUALITY CONTROL SUMMARY FOR ERTEC/TOOELE SURVEY

Analyte: P04 Method: 2P Slope: 0.938 LJD REB  
1.01

Chart #	Lab #	QC #	Type	Date Prepared	Date Analyzed	Target	Found	Fnd./Tar.	F/T Slope	
1	AA004	0001	LEACH	26 FEB	26 FEB	1008	935	.928	.989	
2	AB004	0002	LEACH	26 FEB	1 MAR	1008	985	.977	1.04	
3	AA006	0009	LEACH	5 MAR	5 MAR	1008	874	.867	.924	REB 1.05
4	AA001	0013	LEACH	11 MAR	11 MAR	1008	1031	1.02	1.09	REB 1.09
5	AA005	0023	Water	2 APR	2-3 APR	1008	1030	1.02	1.09	
6	APV002	0024	Water	6 Apr	6 Apr	1008	985	.977	1.04	
7	ABA002	0029	Water	6 Apr	7 Apr	1008	1000	.992	1.06	
8	ABD007	0032	Water	16 APR	16 APR	1008	945	.938	.999	
9	ABD003	0042	Water	30 Apr	30 Apr	1008	1000	.992	1.06	
10	ABP004	0043	Water	3 May	4 May	1008	894	.887	.878	REB 1.05
11	ABQ003	0044	Water	5 May	6 May	1008	1110	1.10	1.09	
	ABP---	0055	Water	12 May	13 May	1008	947	.939	.930	
	ACB003	0056	Leach	4 June	5 June	1008	946	.938	1.00	
	ACG006	0061	Water	25 JUN	25 JUN	1008	913	.906	.897	REB

UBTL QUALITY CONTROL SUMMARY FOR ERTEC/TOOELE SURVEY

Analyte: S04 Method: 2P Slope: 0.934 LJD  
REB  
1.03

Chart #	Lab #	QC #	Type	Date Prepared	Date Analyzed	Target	Found	End./Tar.	F/T Slope	
1	AAAC04	0001	LEACH	26 FEB	26 FEB	1005	1081	1.076	1.15	
2	AAB004	0002	LEACH	26 FEB	1 MAR	1005	1155	1.149	1.23	
3	AAHC06	0009	LEACH	5 MAR	5 MAR	1005	1069	1.049 1.064	1.12 1.14	REKUN 1.12
4	HA1001	0013	LEACH	18 MAR	11 MAR	1005	1021	1.016	1.09	1.016 1.12
5	AAU005	0023	Water	2 APR	2-3 APR	1005	1040	1.035	1.11	
6	AAV002	0024	Water	6 Apr	6 Apr	1005	979	.974	1.04	
7	ABA002	0029	Water	6 Apr	7 Apr	1005	962	.957	1.02	
8	ABD007	0032	Water	16 APR	16 APR	1005	1020	1.015	1.09	
9	ABO003	0042	Water	30 Apr	30 Apr	1000	999	.999	1.07	
10	ABP004	0043	Water	3 May	4 May	1000	976 720,000	.926	.948	1.020 contaminated
11	ABQ003	0044	Water	5 May	6 May	1000	2340	2.340	2.27	
	AP1---	0055	Water	12 May	13 May	1000	920	.920	.897	
	ACT003	0056	Leach	4 June	5 June	1000	967	.967	1.04	
	AC6006	0061	Water	25 JUN	25 JUN	1000	1160	1.160	1.126	(REB)

Analyte:	<u>CYN</u>	Method:	<u>4K</u>	Slope:	<u>0.919</u>
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Analyte: Quia B Method: 30 Slope: .786

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## APPENDIX I

### Summary of Quality Control Data and Field Sample Data

Volatiles by GC/MS (2J)  
Semi-Volatiles by GC/MS (3W)  
Five Explosives by HPLC (2B)  
NG & PETN by HPLC (6B)  
Metals by ICP (3T)  
Metals by GF/AA (7T)  
Mercury by CV/AA (1D)  
Sodium by AA (1M)  
Anions by Ion Chromatography (2P)  
Cyanide by Spectrophotometry (4K)  
Oil and Grease (00)  
Gross Alpha and Gross Beta (30)



Review of Analysis: Volatiles by GC/MS, Method 2J (Water)

Chemist: RWW

Analyte: Benzene (C<sub>6</sub>H<sub>6</sub>), Detection Limit = 1 µg/L, Slope = 1.22

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAW	--	0.16
AAX	--	0.03
ABF	--	0.27
ABU	--	0.34

Analyte: Methyl Bromide (CH<sub>3</sub>BR), Detection Limit = 1 µg/L, Slope = 0.367

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAW	--	--	143%
AAX	--	--	248%
ABF	--	--	No QC
ABU	--	--	478%

Analyte: Chlorobenzene (C<sub>6</sub>H<sub>5</sub>Cl), Detection Limit = 1 µg/L, Slope = 1.39

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAW	--	0.08	66%
AAX	--	--	98%
ABF	--	0.02	No QC
ABU	--	--	73%

Analyte: 1,2-Dichloroethane (C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub>), Detection Limit = 1 µg/L, Slope = 0.767

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAW	--	--	93%
AAX	--	--	110%
ABF	--	--	No QC
ABU	--	0.35	125%

Analyte: Trans-1,2dichloroethene (Tl2DCE), Detection Limit = 1 µg/L, Slope = 0.903

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAW	--	--
AAX	--	--
ABF	--	--
ABU	--	--

Analyte: 1,1,2,2-Tetrachloroethane (TCLEA), Detection Limit = 1 µg/L, Slope 1.01

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAW	--	0.35
AAX	--	--
ABF	--	0.04
ABU	--	--

Analyte: 1,1,1-Trichloroethane (111TCE), Detection Limit = 1 µg/L, Slope = 0.931

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAW	--	--
AAX	--	--
ABF	--	0.82
ABU	--	--

Analyte: Trichloroethene (TRCLE), Detection Limit = 1 µg/L, Slope 1.45

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAW	--	0.88
AAX	--	0.18
ABF	--	0.57
ABU	--	--

Review of Analysis: Volatiles by GC/MS, Method 2J (Water)

Chemist: JMR

Analyte: Benzene (C6H6), Detection Limit = 1 µg/L, Slope = 1.27

<u>Lot</u>	<u>Low</u>	<u>High</u>
ACL	--	0.50

Analyte: Bromo methane (CH3BR), Detection Limit = 1 µg/L, Slope 1.16

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACL	--	--	96%

Analyte: Chlorobenzene (CLC6H5), Detection Limit = 1 µg/L, Slope = 1.03

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACL	--	--	104%

Analyte: 1,2-Dichloroethane (12DCLE), Detection Limit = 1 µg/L, Slope = 1.24

<u>Low</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACL	--	1.6	105%

Analyte: Trans-1,2-dichloroethene (T12DCE), Detection Limit = 1 µg/L, Slope = 1.24

<u>Lot</u>	<u>Low</u>	<u>High</u>
ACL	--	1.0

Analyte: 1,1,2,2-Tetrachloroethane (TCLEA), Detection Limit = 1 µg/L, Slope 1.42

<u>Lot</u>	<u>Low</u>	<u>High</u>
ACL	--	--

Analyte: 1,1,1-Trichloroethane (111TCE), Detection Limit = 1  $\mu\text{g/L}$ , Slope 1.41

<u>Lot</u>	<u>Low</u>	<u>High</u>
ACL	--	291

Analyte: Trichloroethene (TRCLE), Detection Limit = 1  $\mu\text{g/L}$ , Slope = 1.28

<u>Lot</u>	<u>Low</u>	<u>High</u>
ACL	--	1.1

Review of Analysis: Semivolatiles by GC/MS, Method 3W (Soil Leach)

Chemist: JMR

Analyte: Hexachloroethane (CL6ET), Detection Limit = 100 µg/L, Slope = 0.333

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAE	--	--	134%
AAQ	--	--	183%

Analyte: Naphthalene (NAP), Detection Limit = 10 µg/L, Slope = 0.530

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: Nitrobenzene (NB), Detection Limit = 40 µg/L, Slope = 0.516

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: 3,5-Dinitroaniline (35DNA), Detection Limit = 100 µg/L, Slope = 1.19

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAE	--	--	77%
AAQ	--	--	85%

Analyte: 2-Amino-4,6-dinitrotoluene (2A46DT), Detection Limit = 100 µg/L, Slope = 1.38

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	73

Analyte: Fluoranthene (FANT), Detection Limit = 10 µg/L, Slope 1.01

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: 3-Nitrotoluene (3NT), Detection Limit = 50 µg/L, Slope = 0.792

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: Diethylphthalate (DEP), Detection Limit = 10 µg/L, Slope 1.04

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	2.2	5.1
AAQ	--	11

Analyte: Alpha-BHC (ABHC), Detection Limit = 100 µg/L, Slope = 0.983

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: p,p'-DDT (PPDDT), Detection Limit = 10 µg/L, Slope 1.07

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: Dieldrin (DLDRN), Detection Limit = 10 µg/L, Slope = 1.04

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAE	--	--	88%
AAQ	--	--	124%

Analyte: Lindane (LIN), Detection Limit = 100 µg/L, Slope = 1.03

<u>Low</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: Heptachlor (HPCL), Detection Limit = 40 µg/L, Slope = 0.998

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: Aroclor-1016 (PCB016), Detection Limit = 350 µg/L, Slope = 0.925

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: Aroclor-1262 (PCB262), Detection Limit = 500 µg/L, Slope 0.991

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: 2,4-Dimethylphenol (24DMPN), Detection Limit = 100 µg/L, Slope = 0.028

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: 2,4-Dinitrophenol (24DNP), Detection Limit = 150 µg/L, Slope = 1.02

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAE	--	--	79%
AAQ	--	--	78%

Analyte: 2-Methyl-4,6-dinitrophenol (46DN2C), Detection Limit = 100 µg/L, Slope = 1.27

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: Pentachlorophenol (PCP), Detection Limit = 100 µg/L, Slope = 1.14

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	--	--
AAQ	--	--

Analyte: Phenol-D6 (PHEND6), Detection Limit = 15 µg/L, Slope = 0.235

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAE	9.8	280
AAQ	5.5	55



Review of Analysis: Semivolatiles by GC/MS, Method 3W (Soil Leach)

Chemist: RWW

Analyte: Hexachloroethane (CL6ET), Detection Limit = 100 µg/L, Slope = 0.592

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABI	--	--	87%
ACC	--	32	33%

Analyte: Naphthalene (NAP), Detection Limit = 10 µg/L, Slope = 0.76

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: Nitrobenzene (NB), Detection Limit = 40 µg/L, Slope = 0.80

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	0.10
ACC	--	14

Analyte: 3,5-Dinitroaniline (35DNA), Detection Limit = 100 µg/L, Slope = 0.559

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABI	--	--	168%
ACC	--	--	No QC

Analyte: 2-Amino-4,6-dinitrotoluene (2A46DT), Detection Limit = 100 µg/L, Slope = 1.46

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: Fluoranthene (FANT), Detection Limit = 10 µg/L, Slope 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	1.4

Analyte: 3-Nitrotoluene (3NT), Detection Limit = 50 µg/L, Slope = 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	11

Analyte: Diethylphthalate (DEP), Detection Limit = 10 µg/L, Slope 0.99

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	2
ACC	5	20

Analyte: Alpha-BHC (ABHC), Detection Limit = 100 µg/L, Slope = 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: p,p'-DDT (PPDDT), Detection Limit = 10 µg/L, Slope 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: Dieldrin (DLDRN), Detection Limit = 10 µg/L, Slope = 0.70

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABI	--	--	76%
ACC	--	--	No QC

Analyte: Lindane (LIN), Detection Limit = 100 µg/L, Slope = 1.02

<u>Low</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: Heptachlor (HPCL), Detection Limit = 40 µg/L, Slope = 0.76

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: Aroclor-1016 (PCB016), Detection Limit = 350 µg/L, Slope = 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: Aroclor-1262 (PCB262), Detection Limit = 500 µg/L, Slope 0.73

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: 2,4-Dimethylphenol (24DMPN), Detection Limit = 100 µg/L, Slope = 0.60

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	0.71
ACC	--	--

Analyte: 2,4-Dinitrophenol (24DNP), Detection Limit = 150 µg/L, Slope = 1.07

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABI	--	--	121%
ACC	--	--	No QC

Analyte: 2-Methyl-4,6-dinitrophenol (46DN2C), Detection Limit = 100 µg/L, Slope = 1.08

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: Pentachlorophenol (PCP), Detection Limit = 100 µg/L, Slope = 1.02

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Analyte: Phenol-D6 (PHEND6), Detection Limit = 15 µg/L, Slope = 0.252

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ACC	--	--

Review of Analysis: Semivolatiles by GC/MS, Method 3W (Water)

Chemist: JMR

Analyte: Hexachloroethane (CL6ET), Detection Limit = 20 µg/L, Slope = 0.333

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAZ	--	--	176%

Analyte: Naphthalene (NAP), Detection Limit = 2 µg/L, Slope = 0.530

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Nitrobenzene (NB), Detection Limit = 8 µg/L, Slope = 0.516

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: 3,5-Dinitroaniline (35DNA), Detection Limit = 20 µg/L, Slope = 1.19

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAZ	--	--	115%

Analyte: 2-Amino-4,6-dinitrotoluene (2A46DT), Detection Limit = 20 µg/L, Slope = 1.38

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Fluoranthene (FANT), Detection Limit = 2 µg/L, Slope 1.01

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: 3-Nitrotoluene (3NT), Detection Limit = 10 µg/L, Slope = 0.792

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Diethylphthalate (DEP), Detection Limit = 2 µg/L, Slope 1.04

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	<0.1	0.7

Analyte: Alpha-BHC (ABHC), Detection Limit = 20 µg/L, Slope = 0.983

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: p,p'-DDT (PPDDT), Detection Limit = 2 µg/L, Slope 1.07

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Dieldrin (DLDRN), Detection Limit = 2 µg/L, Slope = 1.04

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAZ	--	--	87%

Analyte: Lindane (LIN), Detection Limit = 20 µg/L, Slope = 1.03

<u>Low</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Heptachlor (HPCL), Detection Limit = 8 µg/L, Slope = 0.998

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Aroclor-1016 (PCB016), Detection Limit = 70 µg/L, Slope = 0.925

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Aroclor-1262 (PCB262), Detection Limit = 20 µg/L, Slope 0.991

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: 2,4-Dimethylphenol (24DMPN), Detection Limit = 20 µg/L, Slope = 0.028

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: 2,4-Dinitrophenol (24DNP), Detection Limit = 30 µg/L, Slope = 1.02

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAZ	--	1.6	59%

Analyte: 2-Methyl-4,6-dinitrophenol (46DN2C), Detection Limit = 20 µg/L, Slope = 1.27

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Pentachlorophenol (PCP), Detection Limit = 20 µg/L, Slope = 1.14

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	--

Analyte: Phenol-D6 (PHEND6), Detection Limit = 3 µg/L, Slope = 0.235

<u>Lot</u>	<u>Low</u>	<u>High</u>
AAZ	--	1.4

Review of Analysis: Semivolatiles by GC/MS, Method 3W (Water)

Chemist: RWW

Analyte: Hexachloroethane (CL6ET), Detection Limit = 20 µg/L, Slope = 0.592

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABI	--	--	87%
ABT	--	--	61%
ACN	--	--	86%

Analyte: Naphthalene (NAP), Detection Limit = 2 µg/L, Slope = 0.76

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	0.8
ABT	--	--
ACN	--	--

Analyte: Nitrobenzene (NB), Detection Limit = 8 µg/L, Slope = 0.80

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	--

Analyte: 3,5-Dinitroaniline (35DNA), Detection Limit = 20 µg/L, Slope = 0.559

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABI	--	--	168%
ABT	--	--	41%
ACN	--	--	No QC

Analyte: 2-Amino-4,6-dinitrotoluene (2A46DT), Detection Limit = 20 µg/L, Slope = 1.46

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	19
ABT	--	--
ACN	--	--



Analyte: Fluoranthene (FANT), Detection Limit = 2 µg/L, Slope 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	0.3
ABT	--	--
ACN	--	--

Analyte: 3-Nitrotoluene (3NT), Detection Limit = 10 µg/L, Slope = 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	--

Analyte: Diethylphthalate (DEP), Detection Limit = 2 µg/L, Slope 0.99

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	0.9
ABT	--	--
ACN	--	0.3

Analyte: Alpha-BHC (ABHC), Detection Limit = 20 µg/L, Slope = 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	--

Analyte: p,p'-DDT (PPDDT), Detection Limit = 2 µg/L, Slope 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	1.8
ABT	--	--
ACN	--	--

Analyte: Dieldrin (DLDRN), Detection Limit = 2 µg/L, Slope = 0.70

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABI	--	0.3	76%
ABT	--	--	105%
ACN	--	0.4	83%

Analyte: Lindane (LIN), Detection Limit = 20 µg/L, Slope = 1.02

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	--

Analyte: Heptachlor (HPCL), Detection Limit = 8 µg/L, Slope = 0.76

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	--

Analyte: Aroclor-1016 (PCB016), Detection Limit = 70 µg/L, Slope = 1.00

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	--

Analyte: Aroclor-1262 (PCB262), Detection Limit = 100 µg/L, Slope 0.73

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	--

Analyte: 2,4-Dimethylphenol (24DMPN), Detection Limit = 20 µg/L, Slope = 0.60

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	190
ABT	--	--
ACN	--	14

Analyte: 2,4-Dinitrophenol (24DNP), Detection Limit = 30 µg/L, Slope = 1.07

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABI	--	--	121%
ABT	--	--	48%
ACN	--	--	No QC

Analyte: 2-Methyl-4,6-dinitrophenol (46DN2C), Detection Limit = 20 µg/L, Slope = 1.08

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	22

Analyte: Pentachlorophenol (PCP), Detection Limit = 20 µg/L, Slope = 1.02

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	--
ABT	--	--
ACN	--	--

Analyte: Phenol-D6 (PHEND6), Detection Limit = 3 µg/L, Slope = 0.252

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABI	--	620
ABT	--	--
ACN	--	11

Review of Analysis: 5 Explosives by HPLC, Method 2B (Soil Leach)

Chemist: RMI

Analyte: 2,4-Dinitrotoluene (24DNT), Detection Limit = 10 µg/L, Slope = 0.739

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAD	--	--	43%
AAO	--	10	78%
AAP	--	50	45%
ABH	--	--	90%
ACD	--	--	67%

Analyte: 2,6-Dinitrotoluene (26DNT), Detection Limit = 15 µg/L, Slope = 0.741

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAD	--	--	33%
AAO	--	--	62%
AAP	--	<98	26%
ABH	--	--	89%
ACD	--	--	65%

Analyte: 2,4,6-Trinitrotoluene (246TNT), Detection Limit = 10 µg/L, Slope = 0.852

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAD	--	--	54%
AAO	--	94	76%
AAP	--	29	82%
ABH	--	--	85%
ACD	--	--	94%

Analyte: Tetryl (TETRYL), Detection Limit = 5 µg/L, Slope = 0.897

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAD	--	--	65%
AAO	--	--	24%
AAP	--	--	61%
ABH	--	--	48%
ACD	--	--	99%

Analyte: RDX (RDX), Detection Limit = 5 µg/L, Slope = 0.940

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAD	--	--	116%
AAO	--	260	54%
AAP	--	1100	48%
ABH	--	--	67%
ACD	--	--	186%

Review of Analysis: 5 Explosives by HPLC, Method 2B (Water)

Chemist: RMI

Analyte: 2,4-Dinitrotoluene (24DNT), Detection Limit = 2 µg/L, Slope = 0.739

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABC	--	--	82%
ABH	--	--	90%
ABV	--	--	78%
ACI	--	--	55%

Analyte: 2,6-Dinitrotoluene (26DNT), Detection Limit = 3 µg/L, Slope = 0.741

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABC	--	--	76%
ABH	--	--	89%
ABV	--	--	78%
ACI	--	--	48%

Analyte: 2,4,6-Trinitrotoluene (246TNT), Detection Limit = 2 µg/L, Slope = 0.852

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABC	--	--	96%
ABH	--	--	85%
ABV	--	--	80%
ACI	--	<3.0	89%

Analyte: Tetryl (TETRYL), Detection Limit = 1 µg/L, Slope = 0.897

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABC	--	--	100%
ABH	--	<2.8	48%
ABV	--	--	48%
ACI	--	--	79%

Analyte: RDX (RDX), Detection Limit = 1 µg/L, Slope = 0.940

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABC	--	13	66%
ABH	--	<3.6	67%
ABV	--	--	46%
ACI	--	--	48%

Review of Analysis: NG & PETN by HPLC, Method 6B (Soil Leach)

Chemist: RMI

Analyte: Nitroglycerine (NG), Detection Limit = 100 µg/L, Slope = 0.643

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAC	--	--	112%
AAM	--	--	54%
AAN	--	--	104%
ABG	--	--	Decomposed
ACE	--	--	156%

Analyte: Pentaerythritoltetranitrate (PETN), Detection Limit = 25 µg/L,  
Slope = 1.08

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAC	--	17	70%
AAM	--	--	89%
AAN	--	--	56%
ABG	--	--	Decomposed
ACE	--	--	82%

Review of Analysis: NG & PETN by HPLC, Method 6B (Water)

Chemist: RMI

Analyte: Nitroglycerine (NG), Detection Limit = 20 µg/L, Slope = 0.643

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABB	--	--	127%
ABG	--	--	Decomposed
ABW	--	--	35%
ACH	--	--	132%

Analyte: Pentaerythritoltetranitrate (PETN), Detection Limit = 5 µg/L, Slope = 1.08

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABB	--	--	77%
ABG	--	--	Decomposed
ABW	--	1.5	93%
ACH	--	--	69%



Review of Analysis: Metals by ICP, Method 3T (Soil Leach)

Chemist: DER/RK

Analyte: Arsenic (AS), Detection Limit = 300 µg/L, Slope = 0.973

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	12	84%
AAT	--	30	94%
ABL	--	20	61%
ACJ	75	150	100%

Analyte: Beryllium (BE), Detection Limit = 3 µg/L, Slope = 1.06

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	--	187%
AAT	--	--	112%
ABL	--	--	187%
ACJ	0.5	1.1	187%

Analyte: Cadmium (CD), Detection Limit = 30 µg/L, Slope = 0.964

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	--	96%
AAT	--	--	89%
ABL	--	--	103%
ACJ	2	7	103%

Analyte: Chromium (CR), Detection Limit = 25 µg/L, Slope = 0.967

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	23	83%
AAT	6	22	95%
ABL	--	32	83%
ACJ	2	9	103%

Analyte: Copper (CU), Detection Limit = 30 µg/L, Slope = 1.07

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	14	97%
AAT	--	17	85%
ABL	--	31	157%
ACJ	2	11	78%

Analyte: Lead (PB), Detection Limit = 150 µg/L, Slope 0.928

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	44	104%
AAT	39	140	100%
ABL	--	98	86%
ACJ	10	28	97%

Analyte: Nickel (NI), Detection Limit = 100 µg/L, Slope = 0.931

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	1	86%
AAT	--	--	73%
ABL	--	17	75%
ACJ	7	23	86%

Analyte: Silver (AG), Detection Limit = 40 µg/L, Slope 0.929

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	19	51%
AAT	--	--	98%
ABL	--	--	58
ACJ	--	--	27%

Analyte: Zinc (ZN), Detection Limit = 100 µg/L, Slope = 0.832

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAG	--	1100	No Recovery
AAT	9	63	86%
ABL	24	110	90%
ACJ	16	74	96%

Review of Analysis: Metals by ICP, Method 3T (Water)

Chemist: DER/RK

Analyte: Arsenic (AS), Detection Limit = 60 µg/L, Slope = 0.973

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	--	230	61%
ACA	--	570	95%
ACJ	--	110	100%

Analyte: Beryllium (BE), Detection Limit = 0.5 µg/L, Slope = 1.06

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	--	2.4	187%
ACA	--	3.2	150%
ACJ	--	0.9	187%

Analyte: Cadmium (CD), Detection Limit = 6 µg/L, Slope = 0.964

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	--	31	103%
ACA	--	14	85%
ACJ	--	6	103%

Analyte: Chromium (CR), Detection Limit = 5 µg/L, Slope = 0.967

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	11	310	83%
ACA	--	45	83%
ACJ	2	39	103%

Analyte: Copper (CU), Detection Limit = 6 µg/L, Slope = 1.07

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	2	57	157%
ACA	--	21	94%
ACJ	--	16	78%

Analyte: Lead (PB), Detection Limit = 30 µg/L, Slope 0.928

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	39	1100	86%
ACA	--	110	79%
ACJ	--	72	97%

Analyte: Nickel (NI), Detection Limit = 20 µg/L, Slope = 0.931

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	--	25	75%
ACA	1	39	75%
ACJ	4	490	86%

Analyte: Silver (AG), Detection Limit = 8 µg/L, Slope 0.929

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	--	28	58%
ACA	--	46	54%, 256%
ACJ	--	--	27%

Analyte: Zinc (ZN), Detection Limit = 20 µg/L, Slope = 0.832

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABL	8	230	90%
ACA	18	210	102%, 84%
ACJ	16	57	96%

Review of Analysis: Metals by GF/AA, Method 7T (Soil Leach and Water)

Chemist: CLM

Analyte: Arsenic (AS), Detection Limit = 7 µg/L, Slope = 0.913

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAI	--	--	85%
AAS	--	43	87%
ABJ	--	100	79%
ABZ	--	450	76%
ACP	--	110	111%

Analyte: Nickel (NI), Detection Limit = 5 µg/L, Slope = 0.94

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAI	--	0.5	277%
AAS	3.2	50	259%
ABJ	--	45	323%
ABZ	--	15	90%
ACP	--	660	91%

Analyte: Zinc (ZN), Detection Limit = 1 µg/L, Slope = 1.11

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAI	0.6	6.7	235%
AAS	3.0	51	271%
ABJ	--	74	129%
ABZ	0.4	71	157%
ACP	0.8	78	174%

Review of Analysis: Mercury by CV/AA, Method 1D (Soil Leach)

Chemist: CLM

Analyte: Mercury (HG), Detection Limit = 1 ug/L, Slope = 0.915

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAF	--	0.16	94%
AAK	--	0.04	194%
ABE	0.07	0.21	76%

Chemist: DWB

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACF	--	0.06	81%
ACK	0.20	0.20	100%

Review of Analysis: Mercury by CV/AA, Method 1D (Water)

Chemist: CLM

Analyte: Mercury (HG), Detection Limit = 0.2 µg/L, Slope = 0.915

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAV	--	0.37	250%
ABE	0.01	0.07	76%
ABS	--	--	65%

Chemist: DWB

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACK	--	0.07	100%

Review of Analysis: Sodium by AA, Method 1M (Soil Leach and Water)

Chemist: DFC

Analyte: Sodium (NA), Detection Limit = 1000 µg/L, Slope = 0.989

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAJ	6,900	200,000	122%
AAR	2,000	42,000	102%
ABK	30	4,400,000	92%
ABY	28,000	17,000,000	94%
ACQ	40,000	1,400,000	93%



Review of Analysis: Anions by Ion Chromatography, Method 2P (Soil Leach and Water)

Chemist: LJD

Analyte: Chloride (CL), Detection Limit = 1000 µg/L, Slope = 0.971

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAA	160,000	260,000	106%
AAB	5,900	110,000	116%
AAH	900	20,000	112%
AAL	9,000	9,000	114%
AAU	38,000	200,000	107%
AAV	560,000	890,000	113%
ABA	35,000	340,000	104%
ABD	200	>21,000	122%
ABO	>17,000	>17,000	85%
ABP	13,000	>17,000	110%
ABQ	>17,000	>17,000	279%
ACB	>21,000	>21,000	103%
ACG	>17,000	>17,000	105%

Analyte: Fluoride (FL), Detection Limit = 1000 µg/L, Slope = 0.894

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAA	200	300	108%
AAB	600	800	123%
AAH	200	1,200	94%
AAL	300	300	108%
AAU	200	500	129%
AAV	900	1,200	102%
ABA	400	9,600	105%
ABD	100	3,600	106%
ABO	200	3,200	101%
ABP	400	8,400	76%
ABQ	400	2,300	82%
ACB	600	1,500	107%
ACG	400	7,500	84%

Analyte: Nitrate (NO3), Detection Limit = 1000 µg/L, Slope = 1.14

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAA	6,100	9,600	90%
AAB	500	4,400	94%
AAH	400	2,500	106%
AAL	2,600	2,600	101%
AAU	4,600	10,000	114%
AAV	4,200	240,000	90%
ABA	1,300	11,000	138%
ABD	--	>18,000	99%
ABO	40	9,900	95%
ABP	200	7,300	115%
ABQ	8,900	19,000	133%
ACB	100	500	100%
ACG	50	19,000	112%

Analyte: Nitrite (NO2), Detection Limit = 1000 µg/L, Slope = 1.05

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAA	--	--	84%
AAB	--	--	88%
AAH	50	300	95%
AAL	10	10	93%
AAU	30	80	87%
AAV	20	20	92%
ABA	30	30	101%
ABD	30	30	94%
ABO	--	--	91%
ABP	--	--	95%
ABQ	60	60	82%
ACB	30	30	86%
ACG	20	20	92%

Analyte: Phosphate (P04), Detection Limit = 1000 µg/L, Slope = 0.938

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAA	--	--	99%
AAB	100	100	104%
AAH	100	2,200	92%
AAL	90	80	109%
AAU	80	800	109%
AAV	90	90	104%
ABA	70	400	106%
ABD	30	3,600	100%
ABO	70	60	106%
ABP	80	80	88%
ABQ	70	9,700	109%
ACB	100	100	100%
ACG	200	5,800	90%

Analyte Sulfate (S04), Detection Limit = 1000 µg/L, Slope = 0.934

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
AAA	200	800	115%
AAB	4,300	24,000	123%
AAH	700	9,000	114%
AAL	100	100	109%
AAU	21,000	210,000	111%
AAV	110,000	800,000	104%
ABA	16,000	230,000	102%
ABD	1,200	>21,000	109%
ABO	18,000	>19,000	107%
ABP	19,000	>19,000	95%
ABQ	>19,000	>19,000	227%
ACB	17,000	>21,000	104%
ACG	500	>19,000	113%

Review of Analysis: Cyanide by Spectrophotometry, Method 4K (Water)

Chemist: HFL

Analyte: Cyanide (CYN), Detection Limit = 10  $\mu\text{g/L}$ , Slope = 0.919

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ABM	--	32	72%
ABR	--	0.7	65%
ACM	1.0	12	98%

Review of Analysis: Oil and Grease, Method 00 (Water)

Chemist: MEG

Analyte: Oil and Grease (OILGR), Detection Limit = 5000  $\mu\text{g/L}$ , Not Certified

<u>Lot</u>	<u>Low</u>	<u>High</u>
ABN	--	23,000
ABX	--	5,000
ACO	700	1,800

Review of Analysis: Gross Alpha and Gross Beta, Method 30 (Soil Leach)

CEP Laboratory

Analyte: Gross Alpha (ALPGL), Detection Limit = 10 pCi/L, Slope = 0.893

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACR	<11	<11	95%, 86%, 112%
ACS	<11	<11	111%, 112%

Analyte: Gross Beta (BETGL), Detection Limit = 15 pCi/L, Slope = 0.786

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACR	<19	19	123%, 118%, 136%
ACS	<19	19	134%, 136%

Review of Analysis: Gross Alpha and Gross Beta, Method 30 (Water)

CEP Laboratory

Analyte: Gross Alpha (ALPGL), Detection Limit = 2 pCi/L, Slope = 0.893

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACR	<2.2	29	95%, 86%, 112%
ACS	<2.2	<2.2	111%, 112%

Analyte: Gross Beta (BETGL), Detection Limit = 3 pCi/L, Slope = 0.786

<u>Lot</u>	<u>Low</u>	<u>High</u>	<u>QC</u>
ACR	<3.8	46	123%, 118%, 136%
ACS	<3.8	28	134%, 136%